1Q:1

PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

To:

Commissioner
US Department of Commerce
United States Patent and Trademark Office, PCT
2011 South Clark Place Room CP2/5C24
Arlington, VA 22202
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 14 May 2001 (14.05.01)	
International application No. PCT/ZA00/00163	**Applicant's or agent's file reference** INT1010/MAJR
International filing date (day/month/year) 05 September 2000 (05.09.00)	**Priority date** (day/month/year) 07 September 1999 (07.09.99)
Applicant DEW, David, William et al	

1. The designated Office is hereby notified of its election made:

[X] in the demand filed with the International Preliminary Examining Authority on:

16 February 2001 (16.02.01)

[ ] in a notice effecting later election filed with the International Bureau on:

2. The election [X] was [ ] was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO **34, chemin des Colombettes** **1211 Geneva 20, Switzerland** Facsimile No.: (41-22) 740.14.35	Authorized officer Kiwa Mpay Telephone No.: (41-22) 338.83.38

PATENT COOPERATION TREATY

From the INTERNATIONAL SEARCHING AUTHORITY

PCT

To:
McCALLUM RADEMEYER & FREIMOND
P.O. Box 1130
7 Maclyn House, Bordeaux
2125 Randburg
SOUTH AFRICA

PCT/PTO Rec'd 07 MAR 2002

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL SEARCH REPORT OR THE DECLARATION

(PCT Rule 44.1)

	Date of mailing (*day/month/year*) 07/12/2000
Applicant's or agent's file reference INT1010/MAJR	**FOR FURTHER ACTION** See paragraphs 1 and 4 below
International application No. PCT/ZA 00/00163	International filing date (*day/month/year*) 05/09/2000
Applicant BILLITON INTELLECTUAL PROPERTY B.V. et al.	

1. [X] The applicant is hereby notified that the International Search Report has been established and is transmitted herewith.

   Filing of amendments and statement under Article 19:
   The applicant is entitled, if he so wishes, to amend the claims of the International Application (see Rule 46):

   When? The time limit for filing such amendments is normally 2 months from the date of transmittal of the International Search Report; however, for more details, see the notes on the accompanying sheet.

   Where? Directly to the International Bureau of WIPO
   34, chemin des Colombettes
   1211 Geneva 20, Switzerland
   Fascimile No.: (41-22) 740.14.35

   For more detailed instructions, see the notes on the accompanying sheet.

2. [ ] The applicant is hereby notified that no International Search Report will be established and that the declaration under Article 17(2)(a) to that effect is transmitted herewith.

3. [ ] **With regard to the protest** against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that:

   [ ] the protest together with the decision thereon has been transmitted to the International Bureau together with the applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices.

   [ ] no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made.

4. **Further action(s):** The applicant is reminded of the following:

   Shortly after **18 months** from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau as provided in Rules 90*bis*.1 and 90*bis*.3, respectively, before the completion of the technical preparations for international publication.

   Within **19 months** from the priority date, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase until 30 months from the priority date (in some Offices even later).

   Within **20 months** from the priority date, the applicant must perform the prescribed acts for entry into the national phase before all designated Offices which have not been elected in the demand or in a later election within 19 months from the priority date or could not be elected because they are not bound by Chapter II.

Name and mailing address of the International Searching Authority	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Maria Van der Hoeven

Form PCT/ISA/220 (July 1998)

NOTES TO FORM PCT/ISA/220

These Notes are intended to give the basic instructions concerning the filing of amendments under article 19. The Notes are based on the requirements of the Patent Cooperation Treaty, the Regulations and the Administrative Instructions under that Treaty. In case of discrepancy between these Notes and those requirements, the latter are applicable. For more detailed information, see also the PCT Applicant's Guide, a publication of WIPO.

In these Notes, "Article", "Rule", and "Section" refer to the provisions of the PCT, the PCT Regulations and the PCT Administrative Instructions respectively.

INSTRUCTIONS CONCERNING AMENDMENTS UNDER ARTICLE 19

The applicant has, after having received the international search report, one opportunity to amend the claims of the international application. It should however be emphasized that, since all parts of the international application (claims, description and drawings) may be amended during the international preliminary examination procedure, there is usually no need to file amendments of the claims under Article 19 except where, e.g. the applicant wants the latter to be published for the purposes of provisional protection or has another reason for amending the claims before international pbulication. Furthermore, it should be emphasized that provisional protection is available in some States only.

What parts of the international application may be amended?

Under Article 19, only the claims may be amended.

During the international phase, the claims may also be amended (or further amended) under Article 34 before the International Preliminary Examining Authority. The description and drawings may only be amended under Article 34 before the International Examining Authority.

Upon entry into the national phase, all parts of the international application may be amended under Article 28 or, where applicable, Article 41.

When? Within 2 months from the date of transmittal of the international search report or 16 months from the priority date, whichever time limit expires later. It should be noted, however, that the amendments will be considered as having been received on time if they are received by the International Bureau after the expiration of the applicable time limit but before the completion of the technical preparations for international publication (Rule 46.1).

Where not to file the amendments?

The amendments may only be filed with the International Bureau and not with the receiving Office or the International Searching Authority (Rule 46.2).

Where a demand for international preliminary examination has been/is filed, see below.

How? Either by cancelling one or more entire claims, by adding one or more new claims or by amending the text of one or more of the claims as filed.

A replacement sheet must be submitted for each sheet of the claims which, on account of an amendment or amendments, differs from the sheet originally filed.

All the claims appearing on a replacement sheet must be numbered in Arabic numerals. Where a claim is cancelled, no renumbering of the other claims is required. In all cases where claims are renumbered, they must be renumbered consecutively (Administrative Instructions, Section 205(b)).

The amendments must be made in the language in which the international application is to be published.

What documents must/may accompany the amendments?

Letter (Section 205(b)):

The amendments must be submitted with a letter.

The letter will not be published with the international application and the amended claims. It should not be confused with the "Statement under Article 19(1)" (see below, under "Statement under Article 19(1)").

The letter must be in English or French, at the choice of the applicant. However, if the language of the international application is English, the letter must be in English; if the language of the international application is French, the letter must be in French.

The letter must indicate the differences between the claims as filed and the claims as amended. It must, in particular, indicate, in connection with each claim appearing in the international application (it being understood that identical indications concerning several claims may be grouped),whether

(i) the claim is unchanged;

(ii) the claim is cancelled;

(iii) the claim is new;

(iv) the claim replaces one or more claims as filed;

(v) the claim is the result of the division of a claim as filed.

The following examples illustrate the manner in which amendments must be explained in the accompanying letter:

1. [Where originally there were 48 claims and after amendment of some claims there are 51]:
"Claims 1 to 29, 31, 32, 34, 35, 37 to 48 replaced by amended claims bearing the same numbers; claims 30, 33 and 36 unchanged; new claims 49 to 51 added."

2. [Where originally there were 15 claims and after amendment of all claims there are 11]:
"Claims 1 to 15 replaced by amended claims 1 to 11."

3. [Where originally there were 14 claims and the amendments consist in cancelling some claims and in adding new claims]:
"Claims 1 to 6 and 14 unchanged; claims 7 to 13 cancelled; new claims 15, 16 and 17 added." or
"Claims 7 to 13 cancelled; new claims 15, 16 and 17 added; all other claims unchanged."

4. [Where various kinds of amendments are made]:
"Claims 1-10 unchanged; claims 11 to 13, 18 and 19 cancelled; claims 14, 15 and 16 replaced by amended claim 14; claim 17 subdivided into amended claims 15, 16 and 17; new claims 20 and 21 added."

"Statement under article 19(1)" (Rule 46.4)

The amendments may be accompanied by a statement explaining the amendments and indicating any impact that such amendments might have on the description and the drawings (which cannot be amended under Article 19(1)).

The statement will be published with the international application and the amended claims.

It must be in the language in which the international appplication is to be published.

It must be brief, not exceeding 500 words if in English or if translated into English.

It should not be confused with and does not replace the letter indicating the differences between the claims as filed and as amended. It must be filed on a separate sheet and must be identified as such by a heading, preferably by using the words "Statement under Article 19(1)."

It may not contain any disparaging comments on the international search report or the relevance of citations contained in that report. Reference to citations, relevant to a given claim, contained in the international search report may be made only in connection with an amendment of that claim.

Consequence if a demand for international preliminary examination has already been filed

If, at the time of filing any amendments under Article 19, a demand for international preliminary examination has already been submitted, the applicant must preferably, at the same time of filing the amendments with the International Bureau, also file a copy of such amendments with the International Preliminary Examining Authority (see Rule 62.2(a), first sentence).

Consequence with regard to translation of the international application for entry into the national phase

The applicant's attention is drawn to the fact that, where upon entry into the national phase, a translation of the claims as amended under Article 19 may have to be furnished to the designated/elected Offices, instead of, or in addition to, the translation of the claims as filed.

For further details on the requirements of each designated/elected Office, see Volume II of the PCT Applicant's Guide.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference INT1010/MAJR	**FOR FURTHER ACTION** see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/ZA 00/00163	International filing date *(day/month/year)* 05/09/2000	(Earliest) Priority Date *(day/month/year)* 07/09/1999
Applicant BILLITON INTELLECTUAL PROPERTY B.V. et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

[X] It is also accompanied by a copy of each prior art document cited in this report.

1. **Basis of the report**

   a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

      [ ] the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

   b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

      [ ] contained in the international application in written form.
      [ ] filed together with the international application in computer readable form.
      [ ] furnished subsequently to this Authority in written form.
      [ ] furnished subsequently to this Authority in computer readble form.
      [ ] the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
      [ ] the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. [ ] **Certain claims were found unsearchable** (See Box I).
3. [ ] **Unity of invention is lacking** (see Box II).

4. With regard to the **title,**

   [ ] the text is approved as submitted by the applicant.
   [X] the text has been established by this Authority to read as follows:

   RECOVERY OF COPPER FROM COPPER BEARING SULPHIDE MINERALS BY BIOLEACHING WITH CONTROLLED OXYGEN FEED

5. With regard to the **abstract,**

   [X] the text is approved as submitted by the applicant.
   [ ] the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No. 5

   [ ] as suggested by the applicant.
   [ ] because the applicant failed to suggest a figure.
   [X] because this figure better characterizes the invention.

   [ ] None of the figures.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/ZA 00/00163

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C22B3/18 C22B15/00 C22B3/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C22B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, EPO-Internal, PAJ, COMPENDEX

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 2 225 256 A (TOLMIN MINERAL TECHNOLOGIES NV) 30 May 1990 (1990-05-30)	1,7,13, 34-36
Y	page 1; claims 1,3-6,8-13,20-23; figures 1-8	2-5,9, 11,12, 14-33, 37-47
	page 5, line 29 -page 7, line 20	

Y	US 5 007 620 A (EMMETT JR ROBERT C ET AL) 16 April 1991 (1991-04-16)	14,15, 37,38, 42-46
	column 15, line 49 - line 65; figures 1-7,20,21,33 column 19, line 3 - line 12 column 21, line 45 - line 53	

	-/--	

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
30 November 2000	07/12/2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Bombeke, M

4

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5 919 674 A (TUNLEY TREVOR H) 6 July 1999 (1999-07-06) the whole document ---	4,5,9, 11,12, 18-33, 37-43,46
Y	US 5 462 720 A (ARAGONES JUAN L B) 31 October 1995 (1995-10-31) claims 1,7,11-14; figures 1,2 ---	2,3, 18-33, 37-43, 46,47
A	US 5 413 624 A (SHARP JAMES E ET AL) 9 May 1995 (1995-05-09) column 1, line 63 -column 2, line 23; claims 1-3 ---	1,11,12, 16,22-29
A	EP 0 004 431 A (INTEROX CHEMICALS LTD) 3 October 1979 (1979-10-03) page 3, line 26 -page 4, line 31 ---	16-29
Y	FR 2 640 284 A (COMMISSARIAT ENERGIE ATOMIQUE ;OUEST STE INDLE MINERAIS (FR)) 15 June 1990 (1990-06-15) page 4, line 9 - line 30 ---	16-29
Y	WO 97 05292 A (BACTECH AUSTRALIA LTD) 13 February 1997 (1997-02-13) claims 1,2; example 1 -----	4,5

4

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/ZA 00/00163

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
GB 2225256 A	30-05-1990	AU 630889 B	12-11-1992
		AU 4262989 A	12-04-1990
		US 5021069 A	04-06-1991
		ZA 8907627 A	25-09-1991
US 5007620 A	16-04-1991	US 4974816 A	04-12-1990
		US 4732608 A	22-03-1988
		AT 78240 T	15-08-1992
		AU 607901 B	21-03-1991
		AU 6858087 A	13-08-1987
		CA 1329989 A	07-06-1994
		DE 3780452 A	20-08-1992
		DE 3780452 D	20-08-1992
		DE 3780452 T	11-03-1993
		EP 0294387 A	14-12-1988
		ES 2001827 A	16-06-1988
		FI 883604 A	01-08-1988
		GR 870202 A	05-06-1987
		JP 63502796 T	20-10-1988
		NZ 219219 A	26-04-1990
		WO 8704694 A	13-08-1987
		US 5057284 A	15-10-1991
		US 5227136 A	13-07-1993
		ZW 15687 A	28-10-1987
		BR 8700558 A	08-12-1987
		US 4968008 A	06-11-1990
		US 4728082 A	01-03-1988
		ZA 8606691 A	24-06-1987
		ZA 8700817 A	28-10-1987
		ZM 1587 A	28-03-1988
		ZW 11686 A	18-02-1987
US 5919674 A	06-07-1999	AU 714364 B	23-12-1999
		AU 5964798 A	01-10-1998
		CA 2233417 A	27-09-1998
		CN 1210152 A	10-03-1999
		ZA 9802549 A	30-09-1998
US 5462720 A	31-10-1995	AU 672254 B	26-09-1996
		PT 101436 A,B	30-06-1995
		AU 5310494 A	27-07-1995
US 5413624 A	09-05-1995	US 5248329 A	28-09-1993
		US 5221327 A	22-06-1993
		AU 654544 B	10-11-1994
		AU 1583992 A	15-09-1992
		JP 6506503 T	21-07-1994
		WO 9214848 A	03-09-1992
		US 5283192 A	01-02-1994
EP 0004431 A	03-10-1979	AU 528093 B	14-04-1983
		AU 4520479 A	27-09-1979
		BR 7901755 A	20-11-1979
		CA 1122414 A	27-04-1982
		DE 2960763 D	26-11-1981
		FI 790857 A	24-09-1979
		MX 151115 A	03-10-1984
		PT 69331 A	01-04-1979

Form PCT/ISA/210 (patent family annex) (July 1992)

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/ZA 00/00163

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0004431 A		ZA 7901396 A	30-04-1980
FR 2640284 A	15-06-1990	NONE	
WO 9705292 A	13-02-1997	AU 6509396 A	26-02-1997

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

McCALLUM RADEMEYER & FREIMOND
P.O. Box 1130
7 June Avenue, Bordeaux
2125 Randburg
AFRIQUE DU SUD

PTO/PCT Rec'd 07 MAR 2002

PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Rule 71.1)

Date of mailing *(day/month/year)* 21.12.2001		
Applicant's or agent's file reference INT1010/MAJR	**IMPORTANT NOTIFICATION**	
International application No. PCT/ZA00/00163	International filing date *(day/month/year)* 05/09/2000	Priority date *(day/month/year)* 07/09/1999
Applicant BILLITON INTELLECTUAL PROPERTY B.V. et al.		

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.

2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.

3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. **REMINDER**

   The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

   Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

   For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/	Authorized officer
European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016	Dekker, M Tel.+31 70 340-4046

Form PCT/IPEA/416 (July 1992)

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference INT1010/MAJR	**FOR FURTHER ACTION** See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/ZA00/00163	International filing date *(day/month/year)* 05/09/2000	Priority date *(day/month/year)* 07/09/1999
International Patent Classification (IPC) or national classification and IPC C22B3/18		
Applicant BILLITON INTELLECTUAL PROPERTY B.V. et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 5 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 4 sheets.

3. This report contains indications relating to the following items:

   I ☒ Basis of the report
   II ☐ Priority
   III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   IV ☐ Lack of unity of invention
   V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   VI ☐ Certain documents cited
   VII ☐ Certain defects in the international application
   VIII ☒ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
16/02/2001	21.12.2001
Name and mailing address of the international preliminary examining authority: European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016	Authorized officer Bombeke, M Telephone No. +31 70 340 3576

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/ZA00/00163

I. Basis of the report

1. With regard to the **elements** of the international application *(Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17))*:

Description, pages:

1-26 as originally filed

Claims, No.:

1-29 with telefax of 08/10/2001

Drawings, sheets:

1/6-6/6 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

   (Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-29
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-29
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-29
	No:	Claims	

2. Citations and explanations
   see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

The subject-matter of claims 1-26 resp. 27-29 to a method resp. a plant for recovering copper from a sulphide mineral slurry by bioleaching with a supply of feed gas containing more than 21% oxygen by volume is novel and inventive vis-à-vis the cited prior art, in particular document US-A-5007620, because said prior art does not teach nor fairly suggests the provision of a special control feature, which involves controlling the "dissolved" oxygen concentration in the bioleach slurry at a particular level by controlling (in response to the measured dissolved oxygen concentration) the feed gas supply to the bioreactor.

It follows that claims 1-29 meet the criterion of novelty and of inventive step set forth in Articles 33(2) and 33(3) PCT.

Re Item VIII

Certain observations on the international application

Independent claims 1 and 27 do not meet the requirements of Article 6 PCT in that the matter for which protection is sought is not clearly defined. The following functional statements do not enable the skilled person to determine which concrete technical measures are necessary to perform the stated functions:

- claim 1: "controlling" the dissolved oxygen concentration by "controlling" at least one of the following ....
- claim 27: "control mechanism" whereby in response to .... the supply of oxygen .... is "controlled"

Moreover the control measures according to the method of claim 1 are not consistent with the control means defined in claim 27.

Furthermore the reactor part of claim 27 does not include all the features essential to its definition and performance (Article 6 and Rule 6.3(b) PCT), nor is it clear how the bio-reactor is operatively connected with the (undefined) copper recovery system, cf. figure 1 and figure 5.

WS

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference INT1010/MAJR	**FOR FURTHER ACTION** see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/ZA 00/00163	International filing date *(day/month/year)* 05/09/2000	(Earliest) Priority Date *(day/month/year)* 07/09/1999
Applicant BILLITON INTELLECTUAL PROPERTY B.V. et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

[X] It is also accompanied by a copy of each prior art document cited in this report.

1. **Basis of the report**
   a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.
      - [ ] the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).
   b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :
      - [ ] contained in the international application in written form.
      - [ ] filed together with the international application in computer readable form.
      - [ ] furnished subsequently to this Authority in written form.
      - [ ] furnished subsequently to this Authority in computer readble form.
      - [ ] the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
      - [ ] the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. [ ] **Certain claims were found unsearchable** (See Box I).
3. [ ] **Unity of invention is lacking** (see Box II).

4. With regard to the **title,**
   - [ ] the text is approved as submitted by the applicant.
   - [X] the text has been established by this Authority to read as follows:

   RECOVERY OF COPPER FROM COPPER BEARING SULPHIDE MINERALS BY BIOLEACHING WITH CONTROLLED OXYGEN FEED

5. With regard to the **abstract,**
   - [X] the text is approved as submitted by the applicant.
   - [ ] the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No. 5
   - [ ] as suggested by the applicant.
   - [ ] because the applicant failed to suggest a figure.
   - [X] because this figure better characterizes the invention.
   - [ ] None of the figures.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/ZA 00/00163

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C22B3/18 C22B15/00 C22B3/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C22B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, EPO-Internal, PAJ, COMPENDEX

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 2 225 256 A (TOLMIN MINERAL TECHNOLOGIES NV) 30 May 1990 (1990-05-30)	1,7,13, 34-36
Y	page 1; claims 1,3-6,8-13,20-23; figures 1-8	2-5,9, 11,12, 14-33, 37-47
	page 5, line 29 -page 7, line 20 ---	
Y	US 5 007 620 A (EMMETT JR ROBERT C ET AL) 16 April 1991 (1991-04-16) column 15, line 49 - line 65; figures 1-7,20,21,33 column 19, line 3 - line 12 column 21, line 45 - line 53 --- -/--	14,15, 37,38, 42-46

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
30 November 2000	07/12/2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Bombeke, M

4

Form PCT/ISA/210 (second sheet) (July 1992)

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5 919 674 A (TUNLEY TREVOR H) 6 July 1999 (1999-07-06) the whole document ---	4,5,9, 11,12, 18-33, 37-43,46
Y	US 5 462 720 A (ARAGONES JUAN L B) 31 October 1995 (1995-10-31) claims 1,7,11-14; figures 1,2 ---	2,3, 18-33, 37-43, 46,47
A	US 5 413 624 A (SHARP JAMES E ET AL) 9 May 1995 (1995-05-09) column 1, line 63 -column 2, line 23; claims 1-3 ---	1,11,12, 16,22-29
A	EP 0 004 431 A (INTEROX CHEMICALS LTD) 3 October 1979 (1979-10-03) page 3, line 26 -page 4, line 31 ---	16-29
Y	FR 2 640 284 A (COMMISSARIAT ENERGIE ATOMIQUE ;OUEST STE INDLE MINERAIS (FR)) 15 June 1990 (1990-06-15) page 4, line 9 - line 30 ---	16-29
Y	WO 97 05292 A (BACTECH AUSTRALIA LTD) 13 February 1997 (1997-02-13) claims 1,2; example 1 -----	4,5

4

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/ZA 00/00163

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
GB 2225256 A	30-05-1990	AU 630889 B	12-11-1992
		AU 4262989 A	12-04-1990
		US 5021069 A	04-06-1991
		ZA 8907627 A	25-09-1991
US 5007620 A	16-04-1991	US 4974816 A	04-12-1990
		US 4732608 A	22-03-1988
		AT 78240 T	15-08-1992
		AU 607901 B	21-03-1991
		AU 6858087 A	13-08-1987
		CA 1329989 A	07-06-1994
		DE 3780452 A	20-08-1992
		DE 3780452 D	20-08-1992
		DE 3780452 T	11-03-1993
		EP 0294387 A	14-12-1988
		ES 2001827 A	16-06-1988
		FI 883604 A	01-08-1988
		GR 870202 A	05-06-1987
		JP 63502796 T	20-10-1988
		NZ 219219 A	26-04-1990
		WO 8704694 A	13-08-1987
		US 5057284 A	15-10-1991
		US 5227136 A	13-07-1993
		ZW 15687 A	28-10-1987
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		ZA 8606691 A	24-06-1987
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US 5919674 A	06-07-1999	AU 714364 B	23-12-1999
		AU 5964798 A	01-10-1998
		CA 2233417 A	27-09-1998
		CN 1210152 A	10-03-1999
		ZA 9802549 A	30-09-1998
US 5462720 A	31-10-1995	AU 672254 B	26-09-1996
		PT 101436 A,B	30-06-1995
		AU 5310494 A	27-07-1995
US 5413624 A	09-05-1995	US 5248329 A	28-09-1993
		US 5221327 A	22-06-1993
		AU 654544 B	10-11-1994
		AU 1583992 A	15-09-1992
		JP 6506503 T	21-07-1994
		WO 9214848 A	03-09-1992
		US 5283192 A	01-02-1994
EP 0004431 A	03-10-1979	AU 528093 B	14-04-1983
		AU 4520479 A	27-09-1979
		BR 7901755 A	20-11-1979
		CA 1122414 A	27-04-1982
		DE 2960763 D	26-11-1981
		FI 790857 A	24-09-1979
		MX 151115 A	03-10-1984
		PT 69331 A	01-04-1979

Form PCT/ISA/210 (patent family annex) (July 1992)

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/ZA 00/00163

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0004431 A		ZA 7901396 A	30-04-1980
FR 2640284 A	15-06-1990	NONE	
WO 9705292 A	13-02-1997	AU 6509396 A	26-02-1997

Form PCT/ISA/210 (patent family annex) (July 1992)

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PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference INT1010/MAJR	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/ZA00/00163	International filing date (*day/month/year*) 05/09/2000	Priority date (*day/month/year*) 07/09/1999
International Patent Classification (IPC) or national classification and IPC C22B3/18		
Applicant BILLITON INTELLECTUAL PROPERTY B.V. et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 5 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 4 sheets.

3. This report contains indications relating to the following items:

   I ☒ Basis of the report
   II ☐ Priority
   III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   IV ☐ Lack of unity of invention
   V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   VI ☐ Certain documents cited
   VII ☐ Certain defects in the international application
   VIII ☒ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
16/02/2001	21.12.2001
Name and mailing address of the international preliminary examining authority: European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016	Authorized officer Bombeke, M Telephone No. +31 70 340 3576

Form PCT/IPEA/409 (cover sheet) (January 1994)

I. Basis of the report

1. With regard to the **elements** of the international application *(Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17))*:

   Description, pages:

   1-26 as originally filed

   Claims, No.:

   1-29 with telefax of 08/10/2001

   Drawings, sheets:

   1/6-6/6 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

   These elements were available or furnished to this Authority in the following language: , which is:

   - ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
   - ☐ the language of publication of the international application (under Rule 48.3(b)).
   - ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

   - ☐ contained in the international application in written form.
   - ☐ filed together with the international application in computer readable form.
   - ☐ furnished subsequently to this Authority in written form.
   - ☐ furnished subsequently to this Authority in computer readable form.
   - ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
   - ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

   - ☐ the description, pages:
   - ☐ the claims, Nos.:

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

   (Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-29
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-29
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-29
	No:	Claims	

2. Citations and explanations
   see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

The subject-matter of claims 1-26 resp. 27-29 to a method resp. a plant for recovering copper from a sulphide mineral slurry by bioleaching with a supply of feed gas containing more than 21% oxygen by volume is novel and inventive vis-à-vis the cited prior art, in particular document US-A-5007620, because said prior art does not teach nor fairly suggests the provision of a special control feature, which involves controlling the "dissolved" oxygen concentration in the bioleach slurry at a particular level by controlling (in response to the measured dissolved oxygen concentration) the feed gas supply to the bioreactor.

It follows that claims 1-29 meet the criterion of novelty and of inventive step set forth in Articles 33(2) and 33(3) PCT.

Re Item VIII

Certain observations on the international application

Independent claims 1 and 27 do not meet the requirements of Article 6 PCT in that the matter for which protection is sought is not clearly defined. The following functional statements do not enable the skilled person to determine which concrete technical measures are necessary to perform the stated functions:

- claim 1: "controlling" the dissolved oxygen concentration by "controlling" at least one of the following ....
- claim 27: "control mechanism" whereby in response to .... the supply of oxygen .... is "controlled"

Moreover the control measures according to the method of claim 1 are not consistent with the control means defined in claim 27.

Furthermore the reactor part of claim 27 does not include all the features essential to its definition and performance (Article 6 and Rule 6.3(b) PCT), nor is it clear how the bio-reactor is operatively connected with the (undefined) copper recovery system, cf. figure 1 and figure 5.

CLAIMS

1. A method of recovering copper from a copper bearing sulphide mineral slurry which includes the steps of:

(a) subjecting the slurry in a reactor to a bioleaching process at a temperature in excess of 40°C;

(b) supplying a feed gas which contains in excess of 21% oxygen by volume, to the slurry;

(c) controlling the dissolved oxygen concentration in the slurry at a level of from 0.2×10^{-3} kg/m^3 to 10×10^{-3} kg/m^3 by controlling at least one of the following: the oxygen content of the feed gas; the feed gas supply rate; the rate of feed of the slurry to the reactor; and

(d) recovering copper from a bioleach residue of the bioleaching process.

2. A method according to claim 1 which includes the step of pre-leaching the slurry prior to the bioleaching process of step (a).

3. A method according to claim 2 wherein the pre-leaching is effected using an acidic solution of copper and ferric sulphate.

4. A method according to any one of claims 1 to 3 which includes the step of removing ferric arsenate from the bioleach residue before step (d).

5. A method according to claim 4 wherein the ferric arsenate is removed by precipitation.

6. A method according to any one of claims 1 to 5 wherein the bioleach residue is subjected to a neutralisation step which produces carbon dioxide which is fed to the feed gas of step (b) or directly to the slurry.

7. A method according to any one of claims 1 to 6 wherein in step (d) copper is recovered using a solvent extraction and electrowinning process.

8. A method according to claim 7 wherein oxygen generated during the copper electrowinning is fed to the feed gas of step (b), or directly to the slurry.

9. A method according to claim 7 or 8 wherein raffinate, produced during the solvent extraction, is supplied to at least one of the following: the bioleaching process of step (a), and an external heap leach process.

10. A method according to any one of claims 7 to 9 wherein oxygen generated during the electrowinning process is fed to the feed gas of step (b) or directly to the slurry.

11. A method according to any one of claims 1 to 10 wherein the said slurry contains at least one of the following: arsenical copper sulphides, and copper bearing sulphide minerals refractory to mesophile leaching.

12. A method according to claim 11 wherein the said slurry contain chalcopyrite concentrates.

13. A method according to any one of claims 1 to 12 wherein the feed gas contains in excess of 85% oxygen by volume.

14. A method according to any one of claims 1 to 13 which includes the step of controlling the carbon content of the slurry.

15. A method according to any one of claims 1 to 14 which includes the step of controlling the carbon dioxide content of the feed gas in the range of from 0.5% to 5.0% by volume.

16. A method according to any one of claims 1 to 15 wherein the bioleaching is carried out at a temperature in the range of from 40°C to 100°C.

17. A method according to claim 16 wherein the said temperature is in the range of from 60°C to 85°C.

18. A method according to any one of claims 1 to 15 which includes the step of bioleaching the slurry at a temperature of up to 45°C using mesophile microorganisms.

19. A method according to claim 18 wherein the microorganisms are selected from the following genus groups: *Acidithiobacillus; Thiobacillus; Leptosprillum; Ferromicrobium;* and *Acidiphilium.*

20. A method according to claim 18 or 19 wherein the said microorganisms are selected from the following species: *Acidithiobacillus caldus (Thiobacillus caldus); Acidithiobacillus thiooxidans (Thiobacillus thiooxidans); Acidithiobacillus ferrooxidans (Thiobacillus ferrooxidans); Acidithiobacillus acidophilus (Thiobacillus acidophilus); Thiobacillus prosperus; Leptospirillum ferrooxidans; Ferromicrobium acidophilus;* and *Acidiphilium cryptum.*

21. A method according to any one of claims 1 to 15 which includes the step of bioleaching the slurry at a temperature of from 45°C to 60°C using moderate thermophile microorganisms.

22. A method according to claim 21 wherein the microorganisms are selected from the following genus groups: *Acidithiobacillus* (formerly *Thiobacillus); Acidimicrobium; Sulfobacillus; Ferroplasma (Ferriplasma);* and *Alicyclobacillus.*

23. A method according to claim 21 or 22 wherein the said microorganisms are selected from the following species: *Acidithiobacillus caldus* (formerly *Thiobacillus caldus); Acidimicrobium ferrooxidans; Sulfobacillus acidophilus; Sulfobacillus disulfidooxidans; Sulfobacillus thermosulfidooxidans; Ferroplasma acidarmanus; Thermoplasma acidophilum;* and *Alicyclobacillus acidocaldrius.*

24. A method according to claim 17 which includes the step of bioleaching the slurry at a temperature of from 60°C to 85°C using thermophilic microorganisms.

25. A method according to claim 24 wherein the microorganisms are selected from the following genus groups: *Acidothermus; Sulfolobus; Metallosphaera; Acidianus; Ferroplasma (Ferriplasma); Thermoplasma;* and *Picrophilus.*

26. A method according to claim 24 or 25 wherein the said microorganisms are selected from the following species: *Sulfolobus metallicus; Sulfolobus acidocaldarius; Sulfolobus thermosulfidooxidans; Acidianus infernus; Metallosphaera sedula; Ferroplasma acidarmanus; Thermoplasma acidophilum; Thermoplasma volcanium;* and *Picrophilus oshimae.*

27. A plant for recovering copper from a copper bearing sulphide mineral slurry which includes a reactor vessel, a source which feeds a copper bearing sulphide mineral slurry to the vessel wherein a bioleaching process is carried out at a temperature in excess of 40°C, an oxygen source which supplies oxygen in the form of oxygen enriched gas or substantially pure oxygen to the slurry, a device which measures the dissolved oxygen concentration in the slurry in the vessel, a control mechanism whereby, in response to the said measured dissolved oxygen concentration, the supply of oxygen from the oxygen source to the slurry is controlled to achieve a dissolved oxygen concentration in the slurry of from 0.2×10^{-3} kg/m³ to 10×10^{-3} kg/m³, and a recovery system which recovers copper from a bioleach residue from the reactor vessel.

28. A plant according to claim 27 wherein the reactor vessel is operated at a temperature in excess of 60°C.

29. A plant according to claim 27 or 28 which includes a pre-leaching stage for leaching the copper bearing sulphide mineral slurry before the slurry is fed to the reactor vessel.

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
15 March 2001 (15.03.2001)

PCT

(10) International Publication Number
WO 01/18269 A1

(51) International Patent Classification[7]: C22B 3/18, 15/00, 3/02

(21) International Application Number: PCT/ZA00/00163

(22) International Filing Date: 5 September 2000 (05.09.2000)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
99/5746 7 September 1999 (07.09.1999) ZA

(71) Applicant *(for all designated States except US)*: BILLITON INTELLECTUAL PROPERTY B.V. [NL/NL]; Mariahoeveplean 6, NL-2509 AA The Hague (NL).

(72) Inventors; and

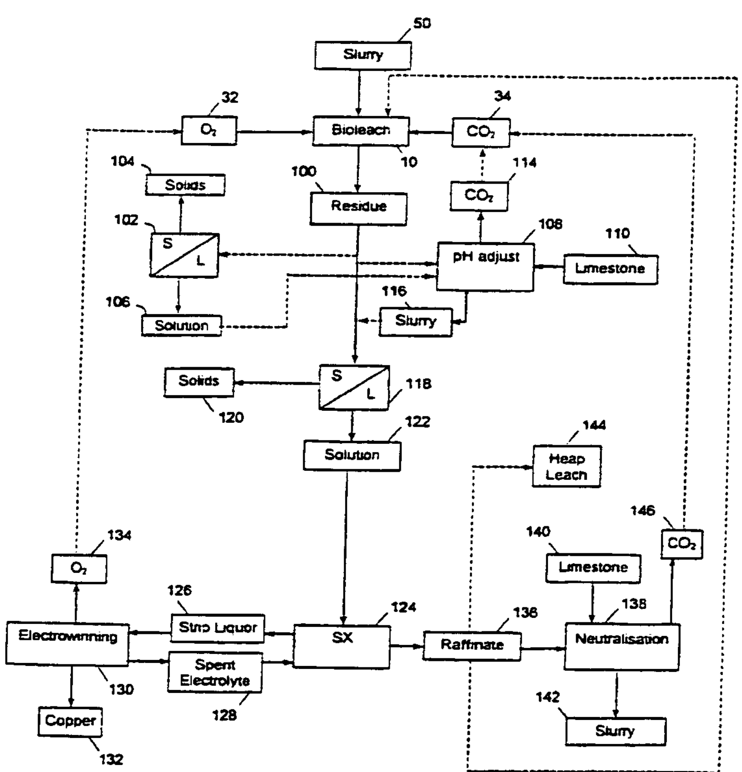
(75) Inventors/Applicants *(for US only)*: DEW, David, William [ZA/ZA]; 33 Alphen Close, Wroxham Road, Paulshof, 2196 Sandton (ZA). BASSON, Petrus [ZA/ZA]; 10 Lourie's Loft, Perm Street, Sonneglans, 2194 Randburg (ZA). MILLER, Deborah, Maxine [ZA/ZA]; 4 Drakenstein, 52 Outspan Road, Sunset Acres, 2001 Johannesburg (ZA).

(74) Agent: RADEMEYER, Montague, Ampie, John: McCallum Rademeyer & Freimond, P.O. Box 1130, 7 Maclyn House, Bordeaux, 2125 Randburg (ZA).

(81) Designated States *(national)*: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ,

[Continued on next page]

(54) Title: RECOVERY OF COPPER FROM COPPER BEARING SULPHIDE MINERALS BY BIOLEACHING WITH CONTROLLED OXYGEN FEED



(57) Abstract: A method of recovering copper from a copper bearing sulphide mineral which includes the steps of subjecting the slurry to a bioleaching process, supplying a feed gas which contains in excess of 21 % oxygen by volume, to the slurry, and recovering copper from a bioleach residue of the bioleaching process.

WO 01/18269 A1

NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) **Designated States** *(regional)*: ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:

— *With international search report.*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

10/069948
JC13 Rec'd PCT/PTO 07 MAR 2002

6/prts

RECOVERY OF COPPER FROM COPPER BEARING SULPHIDE MINERALS BY BIOLEACHING WITH CONTROLLED OXYGEN FEED

BACKGROUND OF THE INVENTION

This invention relates to the recovery of copper from copper bearing sulphide minerals.

Commercial bioleach plants which are currently in operation treating sulphide minerals, typically operate within the temperature range of 40°C to 50°C and rely on sparging air to the bioleach reactors to provide the required oxygen. Operation at this relatively low temperature and the use of air to supply oxygen, limit the rate of sulphide mineral oxidation that can be achieved. For example carrolite and enargite are relatively slow leaching at temperatures below 50°C. and treatment at or below this temperature would result in poor and sub-economic metal extraction.

The use of high temperatures between 50°C and 100°C greatly increases the rate of sulphide mineral leaching.

The solubility of oxygen is however limited at high temperatures and the rate of sulphide mineral leaching becomes limited. In the case of using air for the supply of oxygen, the effect of limited oxygen solubility is such that the rate of sulphide mineral leaching becomes dependent on and is limited by the rate of oxygen transfer from the gas to the liquid phase [1,2].

The bioleaching of secondary copper bearing sulphide minerals is similarly problematic and to the applicant's knowledge no commercial copper bioleach plants are in operation.

More particularly chalcopyrite has long been known to be generally refractory to bioleaching using mesophiles. A major challenge is the leaching of chalcopyrite, on an industrial scale, using thermophilic microorganisms.

SUMMARY OF THE INVENTION

The invention provides a method of recovering copper from a copper bearing sulphide mineral slurry which includes the steps of:

(a) subjecting the slurry to a bioleaching process,

(b) supplying a feed gas which contains in excess of 21% oxygen by volume, to the slurry, and

(c) recovering copper from a bioleach residue of the bioleaching process.

The method may include the step of pre-leaching the slurry prior to the bioleaching process of step (a). The pre-leaching may be effected using an acidic solution of copper and ferric sulphate.

The method may include the step of removing ferric arsenate from the bioleach residue before step (c). The ferric arsenate may be removed by precipitation.

The bioleach residue may be subjected to a neutralisation step which produces carbon dioxide which is fed to the feed gas of step (b), or directly to the slurry.

In step (c) copper may be recovered using a solvent extraction and electrowinning process. Oxygen which is generated during the copper electrowinning may be fed to the feed gas of step (b), or directly to the slurry.

Raffinate, produced by the solvent extraction, may be supplied to at least one of the following: the bioleaching process of step (a), and an external heap leach process.

Oxygen generated during the electrowinning process may be fed to the feed gas of step (b), or directly to the slurry.

The said slurry may contain at least one of the following: arsenical copper sulphides, and copper bearing sulphide minerals which are refractory to mesophile leaching.

The slurry may contain chalcopyrite concentrates.

As used herein the expression "oxygen enriched gas" is intended to include a gas, eg. air, which contains in excess of 21% oxygen by volume. This is an oxygen content greater than the oxygen content of air. The expression "pure oxygen" is intended to include a gas which contains in excess of 85% oxygen by volume.

Preferably the feed gas which is supplied to the slurry contains in excess of 85% oxygen by volume ie. is substantially pure oxygen.

The method may include the step of maintaining the dissolved oxygen concentration in the slurry within a desired range which may be determined by the operating conditions and the type of microorganisms used for leaching. The applicant has established that a lower limit for the dissolved oxygen concentration to sustain microorganism growth and mineral oxidation, is in the range of from 0.2×10^{-3} kg/m^3 to 4.0×10^{-3} kg/m^3. On the other hand if the dissolved oxygen concentration is too high then microorganism growth is inhibited. The upper threshold concentration also depends on the genus and strain of microorganism used in the leaching process and typically is in the range of from 4×10^{-3} kg/m^3 to 10×10^{-3} kg/m^3.

Thus, preferably, the dissolved oxygen concentration in the slurry is maintained in the range of from 0.2×10^{-3} kg/m^3 to 10×10^{-3} kg/m^3.

The method may include the steps of determining the dissolved oxygen concentration in the slurry and, in response thereto, of controlling at least one of the following: the oxygen content of the feed gas, the rate of supply of the feed gas to the slurry, and the rate of feed of slurry to a reactor.

The dissolved oxygen concentration in the slurry may be determined in any appropriate way, e.g. by one or more of the following: by direct measurement of the dissolved oxygen concentration in the slurry, by measurement of the oxygen content in gas above the slurry, and indirectly by measurement of the oxygen content in off-gas from the slurry, taking into account the rate of oxygen supply, whether in gas enriched or pure form, to the slurry, and other relevant factors.

The method may include the step of controlling the carbon content of the slurry. This may be achieved by one or more of the following: the addition of carbon dioxide gas to the slurry, and the addition of other carbonaceous material to the slurry.

The method may extend to the step of controlling the carbon dioxide content of the feed gas to the slurry in the range of from 0.5% to 5% by volume. A suitable figure is of the order of 1% to 1.5% by volume. The level of the carbon dioxide is chosen to maintain high rates of microorganism growth and sulphide mineral oxidation.

The bioleaching process is preferably carried out at an elevated temperature. As stated hereinbefore the bioleaching rate increases with an increase in operating temperature. Clearly the microorganisms which are used for bioleaching are determined by the operating temperature and vice versa. As the addition of oxygen enriched gas or substantially pure oxygen to the slurry has a cost factor it is desirable to operate at a temperature which increases the leaching rate by an amount which more than compensates for the increase in operating cost. Thus, preferably, the bioleaching is carried out at a temperature in excess of 40°C.

The bioleaching may be carried out at a temperature of up to 100°C or more and preferably is carried out at a temperature which lies in a range of from 60°C to 85°C.

In one form of the invention the method includes the step of bioleaching the slurry at a temperature of up to 45°C using mesophile microorganisms. These microorganisms may, for example, be selected from the following genus groups:

Acidithiobacillus (formerly *Thiobacillus*); *Leptosprillum*; *Ferromicrobium*; and *Acidiphilium*.

In order to operate at this temperature the said microorganisms may, for example, be selected from the following species:

Acidithiobacillus caldus (Thiobacillus caldus); *Acidithiobacillus thiooxidans (Thiobacillus thiooxidans)*; *Acidithiobacillus ferrooxidans (Thiobacillus ferrooxidans)*; *Acidithiobacillus acidophilus (Thiobacillus*

acidophilus); Thiobacillus prosperus; Leptospirillum ferrooxidans; Ferromicrobium acidophilus; and *Acidiphilium cryptum.*

If the bioleaching step is carried out at a temperature of from 45°C to 60°C then moderate thermophile microorganisms may be used. These may, for example, be selected from the following genus groups: *Acidithiobacillus* (formerly *Thiobacillus); Acidimicrobium; Sulfobacillus; Ferroplasma (Ferriplasma);* and *Alicyclobacillus.*

Suitable moderate thermophile microorganisms may, for example, be selected from the following species: *Acidithiobacillus caldus* (formerly *Thiobacillus caldus); Acidimicrobium ferrooxidans; Sulfobacillus acidophilus; Sulfobacillus disulfidooxidans; Sulfobacillus thermosulfidooxidans; Ferroplasma acidarmanus; Thermoplasma acidophilum;* and *Alicyclobacillus acidocaldrius.*

It is preferred to operate the leaching process at a temperature in the range of from 60°C to 85°C using thermophilic microorganisms. These may, for example, be selected from the following genus groups: *Acidothermus; Sulfolobus; Metallosphaera; Acidianus; Ferroplasma (Ferriplasma); Thermoplasma;* and *Picrophilus.*

Suitable thermophilic microorganisms may, for example, be selected from the following species: *Sulfolobus metallicus; Sulfolobus acidocaldarius; Sulfolobus thermosulfidooxidans; Acidianus infernus; Metallosphaera sedula; Ferroplasma acidarmanus; Thermoplasma acidophilum; Thermoplasma volcanium;* and *Picrophilus oshimae.*

The slurry may be leached in a reactor tank or vessel which is open to atmosphere or substantially closed. In the latter case vents for off-gas may be provided from the reactor.

According to a different aspect of the invention there is provided a method of recovering copper from a slurry containing copper bearing sulphide minerals which includes the steps of bioleaching the slurry using suitable

microorganisms at a temperature in excess of 40°C, controlling the dissolved oxygen concentration in the slurry within a predetermined range, and recovering copper from a bioleach residue.

The bioleaching may be carried out at a temperature in excess of 60°C.

The dissolved oxygen concentration may be controlled by controlling the addition of gas which contains in excess of 21% oxygen by volume to the slurry.

Preferably the gas contains in excess of 85% by volume.

The bioleach residue may be subjected to a separation step to produce residue solids and solution and the copper may be recovered from the solution in any appropriate way, for example by means of a solvent extraction and electrowinning process.

The invention also extends to a method of enhancing the oxygen mass transfer coefficient from a gas phase to a liquid phase in a copper bearing sulphide mineral slurry which includes the step of supplying a feed gas containing in excess of 21% oxygen by volume to the slurry.

The feed gas preferably contains in excess of 85% oxygen by volume.

The invention further extends to a method of bioleaching an aqueous slurry containing copper bearing sulphide minerals which includes the steps of bioleaching the slurry at a temperature above 40°C and maintaining the dissolved oxygen concentration in the slurry in the range of from 0.2×10^{-3} kg/m^3 to 10×10^{-3} kg/m^3.

The dissolved oxygen concentration may be maintained by supplying gas containing in excess of 21% oxygen by volume to the slurry. The temperature is preferably in the range of from 60°C to 85°C.

The invention further extends to a plant for recovering copper from a copper bearing sulphide mineral slurry which includes a reactor vessel, a source which feeds a copper bearing sulphide mineral slurry to the vessel, an oxygen source, a device which measures the dissolved oxygen concentration in the slurry in the vessel, a control mechanism whereby, in response to the said measure of dissolved oxygen concentration, the supply of oxygen from the oxygen source to the slurry is controlled to achieve a dissolved oxygen concentration in the slurry within a predetermined range, and a recovery system which recovers copper from a bioleach residue from the reactor vessel.

The oxygen may be supplied in the form of oxygen enriched gas or substantially pure oxygen.

The reactor vessel may be operated at a temperature in excess of 60°C and preferably in the range of 60°C to 85°C.

The plant may include a pre-leaching stage for leaching the copper bearing sulphide mineral slurry before the slurry is fed to the reactor vessel. In the pre-leaching stage use may be made of an acidic solution of copper and ferric sulphate.

Various techniques may be used for controlling the supply of oxygen to the slurry and hence for controlling the dissolved oxygen concentration in the slurry at a desired value. Use may for example be made of valves which are operated manually. For more accurate control use may be made of an automatic control system. These techniques are known in the art and are not further described herein.

As has been indicated oxygen and carbon dioxide may be added to the slurry in accordance with predetermined criteria. Although the addition of these materials may be based on expected demand and measurement of other performance parameters, such as iron(II) concentration, it is preferred to make use of suitable measurement probes to sample the actual values of the critical parameters.

For example use may be made of a dissolved oxygen probe to measure the dissolved oxygen concentration in the slurry directly. To achieve this the probe is immersed in the slurry. The dissolved oxygen

concentration may be measured indirectly by using a probe in the reactor off-gas or by transmitting a sample of the off-gas, at regular intervals, to an oxygen gas analyser. Again it is pointed out that measuring techniques of this type are known in the art and accordingly any appropriate technique can be used.

A preferred approach to the control aspect is to utilise one or more probes to measure the dissolved oxygen concentration in the slurry, whether directly or indirectly. The probes produce one or more control signals which are used to control the operation of a suitable valve or valves, eg. solenoid valves, automatically so that the supply of oxygen to an air stream which is being fed to the slurry is varied automatically in accordance with real time measurements of the dissolved oxygen concentration in the slurry.

Although it is preferred to control the addition of oxygen to a gas stream which is fed to the slurry a reverse approach may be adopted in that the oxygen supply rate to the reactor vessel may be maintained substantially constant and the rate of supply of the sulphide mineral slurry to the reactor vessel may be varied to achieve a desired dissolved oxygen concentration.

The invention is not limited to the actual control technique employed and is intended to extend to variations of the aforegoing approaches and to any equivalent process.

The method of invention is of particular benefit to chalcopyrite concentrates, which are more-or-less refractory to leaching at mesophile operating temperatures. The method of the invention therefore opens the door to commercial thermophile leaching of chalcopyrite which to the applicant's knowledge was previously not possible. The added benefits of a high specific reactor sulphide oxidation duty and reduced specific power requirement for oxidation, while still advantageous, are of less significance in this instance.

Additionally copper bearing sulphide flotation concentrates frequently contain chalcocite and the method of the invention is of particular benefit, because chalcocite has a high leaching rate, even at typical mesophile operating temperatures, which is further increased at the higher temperatures used with moderate and extreme thermophiles. Thus the benefits of the invention, including a high specific reactor sulphide oxidation duty and reduced specific power requirement for oxidation, will be particularly beneficial during the

bioleaching of copper bearing sulphide concentrates containing chalcocite, even at typical mesophile operating temperatures.

Copper may be recovered from solution by any appropriate process, for example solvent extraction followed by electrowinning, iron precipitation, or by resin-in-pulp applied to the slurry, followed by electrowinning.

If electrowinning is selected as the production method for copper, the oxygen generated at the anode in the electrowinning process may be used to supplement that used in the bioleach process, reducing the capital and operating costs required for oxygen production.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention is further described by way of examples with reference to the accompanying drawings in which:

Figure 1 is a schematic representation of a portion of a plant in which the invention is carried out,

Figures 2, 3 and 4 shows various results and parameters obtained from operating a bioreactor in the manner described herein with reference to Figure 1, and

Figures 5 and 6 are process flow charts of copper recovery processes according to different forms of the invention.

DESCRIPTION OF PREFERRED EMBODIMENTS

General Principles

The limitation of low oxygen solubility during bioleaching, using air, at high temperatures, which in turn limits the rate of reaction, requires enrichment of the air with oxygen ie. air with an oxygen content greater than 21% by volume, or the use of pure oxygen (defined as being greater than 85% oxygen by volume). The use of oxygen enriched air or pure oxygen overcomes the limited rate of reaction due to oxygen supply constraints, but has two major disadvantages:

a) the provision of oxygen enriched air or pure oxygen is expensive and requires a high utilisation (>60%) of the oxygen to warrant the additional expense [3]; and

b) if the oxygen level in solution becomes too high microorganism growth is prevented and sulphide mineral bioleaching stops [4].

Therefore, in order to realise the benefits of high rates of sulphide mineral leaching at high temperatures in commercial bioleaching plants, the drawbacks of requiring expensive oxygen and the risk of failure if the dissolved oxygen levels become too high must be overcome.

The bioleaching of sulphide minerals at an elevated temperature results in a high rate of sulphide mineral oxidation, but is dependent on the supply of oxygen and carbon dioxide to maintain high rates of sulphide mineral oxidation and of microorganism growth at adequate rates. The absorption of oxygen and carbon dioxide in the bioleaching reactor is limited, in each case, by the rate of mass transfer from the gas phase into the solution phase. For oxygen the rate of oxygen absorption is defined by equation (1) as follows:

$$R = M.(C^* - C_L) \qquad (1)$$

where: R = Oxygen demand as mass (kg) per unit volume (m^3) per unit time(s) ($kg/m^3/s$),

M = Oxygen mass transfer coefficient in reciprocal seconds (s^{-1}),

C^* = Saturated dissolved oxygen concentration as mass (kg) per unit volume (m^3) (kg/m^3), and

C_L = Dissolved oxygen concentration in solution as mass (kg) per unit volume (m^3) (kg/m^3).

The factor ($C^* - C_L$) is referred to as the oxygen driving force. A similar equation may be used to describe the rate of carbon dioxide supply to the solution. If the sulphide mineral oxidation rate is increased the oxygen demand increases proportionately. To meet a higher oxygen demand either the oxygen mass transfer coefficient (M) or the oxygen driving force (C^*-C_L) must be increased.

An increase in the oxygen mass transfer coefficient may be achieved by increasing the power input to the bioleach reactor mixer. This improves gas dispersion in the sulphide mineral slurry. With this approach,

however, an increase in the oxygen mass transfer coefficient of, for example, 40% requires an increase in the power input to the mixer by a factor of as much as 200%, with a commensurate increase in operating costs.

The oxygen driving force may be increased by increasing the saturated dissolved oxygen concentration C^* and reducing the dissolved oxygen content or concentration C_L.

Microorganism population growth is limited or prevented if the dissolved oxygen concentration C^* reaches too high a level. A concentration level above 4×10^{-3} kg/m³ has been found to be detrimental to *Sulfolobus*-like strains. Certain *Acidithiobacillus* strains, however, have been found to be tolerant to dissolved oxygen concentrations of up to 10×10^{-3} kg/m³.

The applicant has established that a lower limit for the dissolved oxygen concentration to sustain microorganism growth and mineral oxidation is in the range of from 0.2×10^{-3} kg/m³ to 4.0×10^{-3} kg/m³. Thus, in order to provide an adequate, or optimum, supply of oxygen, the dissolved oxygen concentration in the sulphide mineral slurry must be monitored and, where appropriate, the addition of oxygen to the sulphide mineral slurry must be controlled in order to maintain the minimum dissolved oxygen concentration in solution at a value of from 0.2×10^{-3} kg/m³ to 4.0×10^{-3} kg/m³.

On the other hand the dissolved oxygen concentration must not exceed an upper threshold value at which microorganism growth is prevented. It is pointed out that the upper threshold concentration depends on the genus and strain of microorganism used in the bioleaching process. A typical upper threshold value is in the range of from 4×10^{-3} kg/m³ to 10×10^{-3} kg/m³.

As has been previously indicated the rate of sulphide mineral oxidation, which can be achieved when operating at a relatively low temperature of the order of from 40°C to 55°C, is limited. In order to increase the rate of oxidation it is desirable to make use of thermophiles and to operate at temperatures in excess of 60°C. Any suitable microorganism capable of operating within this temperature range may be used. The optimum operating temperature is dependent on the genus and type of microorganism used. Thus moderate thermophiles of the type *Sulfobacillus* are suitable for operating at a temperature of up to 65°C. Thermophiles

of the type *Sulfolobus* are suitable for operating at temperatures of from 60°C to at least 85°C. *Sulfolobus metallicus*, for example, shows optimal growth in the temperature range of from 65°C to 70°C.

The applicant has established that the operation of the bioleaching process, using a gas enriched with oxygen, or pure oxygen, as the oxidant, at elevated temperatures of from 40°C to 85°C:

increases the specific sulphide oxidation duty of the reactor considerably;

results in an unexpected and significantly enhanced oxygen mass transfer rate;

increases the oxygen utilisation, providing that the dissolved oxygen concentration is controlled above the point where microorganism growth and mineral oxidation are prevented and below the point at which microorganism growth is inhibited; and

the overall power required for the oxidation of sulphide minerals is significantly reduced.

The method of the invention represents a significant improvement compared to a bioleach operation carried out at a temperature of from 40°C to 45°C with air.

The controlled addition of oxygen enriched air or pure oxygen directly into the bioreactor improves the oxygen utilisation efficiency. The oxygen utilisation for a conventional commercial bioleach plant (at least $100m^3$ in volume) operating at from 40°C to 45°C with air may be expected to achieve a maximum oxygen utilisation factor of from 40% to 50%. Consequently only 40% to 50% of the total mass of oxygen supplied to the bioleach plant is used to oxidise the sulphide minerals. With the method of the invention the oxygen utilisation is significantly higher, of the order of from 60% to 95%. The higher oxygen utilisation is achieved by controlled oxygen addition and results from the enhanced oxygen mass transfer rate and by operating at low dissolved oxygen concentrations in the solution phase.

It will be appreciated that although high oxygen demand in bioleach reactors has come about primarily by the use of higher temperatures, rapidly leaching sulphide minerals at temperatures below 60°C, using mesophile or moderate thermophile microorganisms, will have similarly high oxygen demands. The method of the invention is therefore not restricted to suit thermophiles or extreme thermophiles, but also mesophile and moderate thermophile microorganisms.

Another advantage of using air enriched with oxygen or pure oxygen is that the evaporation losses are reduced, because there is less inert gas removing water vapour from the top of the reactor. This is particularly important in areas where water is scarce or expensive.

In carrying out the method of the invention the temperature of the slurry in the bioleach vessel or reactor may be controlled in any suitable way known in the art. In one example the bioleach reactor is insulated and heating takes place by means of energy which is released by the oxidation of sulphides. The temperature of the slurry is regulated using any suitable cooling system, for example an internal cooling system.

Table 1 shows typical data for specific sulphide oxidation duty and oxygen utilisation, when bioleaching with air at 40°C to 45°C, in two commercial bioreactors, Plant A and Plant B respectively, (greater than 100m³ in volume).

Table 1 Commercial Bioreactor Performance Results

Description	Units	Plant A	Plant B
Reactor temperature	°C	42	40
Reactor operating volume	m^3	471	896
Oxygen utilisation	%	37.9	43.6
Typical dissolved oxygen concentration	mg/l	2.5	2.7
Oxygen mass transfer coefficient	s^{-1}	0.047	0.031
Specific oxygen demand	$kg/m^3/day$	21.6	14.8
Specific sulphide oxidation duty	$kg/m^3/day$	8.9	5.7
Specific power consumption per kg sulphide oxidised	kWh/kgS^{2-}	1.7	1.8

At low temperatures (40°C – 50°C), with air as the inlet gas, which applies to the results for the commercial reactors, Plant A and Plant B, presented in Table 1, the oxygen utilisations achieved are expected and the oxygen mass transfer coefficients (M) correspond to the applicant's design value. The applicant has

determined that if the method of the invention were to be applied to Plant A, the plant performance would be significantly increased, as indicated by the results presented in Table 2.

Table 2 Predicted Improvement In Commercial Bioreactor Performance

	Units	Plant A – typical operation	Plant A - using the method of the invention
Reactor temperature	°C	42	77
Microbial type strain	-	*Acidithiobacillus*	*Sulfolobus*
Inlet gas oxygen content	% by volume	20.9	90.0
Oxygen utilisation	%	37.9	93.0
Typical dissolved oxygen concentration	mg/l	2.5	2.5
Specific oxygen demand	$kg/m^3/day$	21.6	59.5
Specific sulphide oxidation duty	$kg/m^3/day$	8.9	24.5
Specific power consumption per kg sulphide oxidised	kWh/kgS^{2-}	1.7	1.2

The results clearly show the benefit of the invention in achieving higher rates of reaction by the combination of bioleaching at high temperature, adding oxygen enriched gas and by controlling the dissolved oxygen concentration to a predetermined low level (e.g. 0.2×10^{-3} kg/m³ to 4.0×10^{-3} kg/m³). The specific sulphide oxidation duty of the reactor is increased by almost threefold. Clearly the upper dissolved oxygen concentration should not be increased above a value at which microorganism growth is inhibited or stopped.

Even though additional capital for the production of oxygen is required, the savings in reactor and other costs at least offset this additional expense. Additionally, the specific power consumption per kg sulphide oxidised is decreased by approximately one-third. In a plant oxidising 300 tonnes of sulphide per day, the power saving, assuming a power cost of US$0.05 per kWh, would amount to US$2.8 million per annum. The high oxygen utilisation and increased specific sulphide oxidation capacity of the reactor represent in combination a considerable improvement over conventional bioleaching practice conducted at lower temperatures, with oxygen supplied by air.

Bioleaching Plant

Figure 1 of the accompanying drawings shows a bioleaching plant 10 in which bioleaching is carried out, in accordance with the principles of the invention.

The plant 10 includes a bioreactor 12 with an agitator or mixer 14 which is driven by means of a motor and gearbox assembly 16.

In use a tank or vessel 18 of the reactor contains a sulphide mineral slurry 20. An impeller 22 of the agitator is immersed in the slurry and is used for mixing the slurry in a manner which is known in the art.

A probe 24 is immersed in the slurry and is used for measuring the dissolved oxygen concentration in the slurry. A second probe 26, inside the tank 18 above the surface level 28 of the slurry, is used for measuring the carbon dioxide content in the gas 30 above the slurry 20.

An oxygen source 32, a carbon dioxide source 34 and an air source 36 are connected through respective control valves 38, 40 and 42 to a sparging system 44, positioned in a lower zone inside the tank 18, immersed in the slurry 20.

The probe 24 is used to monitor the dissolved oxygen concentration in the sulphide mineral slurry 20 and provides a control signal to a control device 46. The control device controls the operation of the oxygen supply valve 38 in a manner which is known in the art but in accordance with the principles which are described herein in order to maintain a desired dissolved oxygen concentration in the slurry 20.

The probe 26 measures the carbon dioxide content in the gas above the sulphide mineral slurry 20. The probe 26 provides a control signal to a control device 48 which, in turn, controls the operation of the valve 40 in order to control the addition of carbon dioxide from the source 34 to a gas stream flowing to the sparger 44.

The air flow rate from the source 36 to the sparger 44 is controlled by means of the valve 42. Normally the valve is set to provide a more or less constant flow of air from the source 36 to the sparger and the additions of oxygen and carbon dioxide to the air stream are controlled by the valves 38 and 40 respectively. Although this is a preferred approach to adjusting the oxygen and carbon dioxide contents in the air flow to the sparger other techniques can be adopted. For example it is possible, although with a lower degree of preference, to adjust the air stream flow rate and to mix the adjustable air stream with a steady supply of oxygen and a variable supply of carbon dioxide, or vice versa. Another possibility is to have two separate air stream flows to which are added oxygen and carbon dioxide respectively. Irrespective of the technique which is adopted the objective remains the same. namely to control the additions of oxygen and carbon dioxide to the slurry 20.

Slurry 50 is fed from a slurry feed source 52 through a control valve 54 and through an inlet pipe 56 into the interior of the tank 18. The slurry feed rate may be maintained substantially constant, by appropriate adjustment of the valve 54, to ensure that slurry is supplied to the tank 18 at a rate which sustains an optimum leaching rate. The supplies of air, oxygen and carbon dioxide are then regulated, taking into account the substantially constant slurry feed rate, to achieve a desired dissolved oxygen concentration in the slurry 20 in the tank, and a desired carbon dioxide content in the gas 30 above the slurry. Although this is a preferred approach it is apparent that the slurry feed rate could be adjusted, in response to a signal from the probe 24. to achieve a desired dissolved oxygen concentration in the slurry. In other words the rate of oxygen addition to the slurry may be kept substantially constant and the slurry feed rate may be varied according to requirement.

Another variation which can be adopted is to move the probe 24 from a position at which it is immersed in the slurry to a position designated 24A at which it is located in the gas 30 above the level 28. The probe then measures the oxygen contained in the gas above the slurry ie. the bioreactor off-gas. The oxygen content in the off-gas can also be used as a measure to control the dissolved oxygen concentration in the slurry, taking any other relevant factors into account.

Conversely it may be possible to move the carbon dioxide probe 26 (provided it is capable of measuring the dissolved carbon dioxide content) from a position at which it is directly exposed to the gas 30 to a position designated 26A at which it is immersed in the slurry in the tank. The signal produced by the probe at the position 26A is then used, via the control device 48, to control the addition of carbon dioxide from the source 34 to the air stream from the source 36.

Although the carbon dioxide source 34, which provides carbon dioxide in gas form, is readily controllable and represents a preferred way of introducing carbon into the slurry 20, it is possible to add suitable carbonate materials to the slurry 50 before feeding the slurry to the reactor. Carbonate material may also be added directly to the sulphide mineral slurry 20 in the reactor. In other cases though there may be sufficient carbonate in the sulphide mineral slurry so that it is not necessary to add carbon, in whatever form, to the slurry nor to control the carbon content in the slurry.

It is apparent from the aforegoing description which relates to the general principles of the invention that the supply of oxygen to the slurry is monitored and controlled to provide a desired dissolved oxygen concentration level in the slurry 20. This can be done in a variety of ways eg. by controlling one or more of the following in an appropriate manner namely: the slurry feed rate, the air flow rate from the source 36, the oxygen flow rate from the source 32, and any variation of the aforegoing.

The carbon dioxide flow rate is changed in accordance with the total gas flow rate to the sparger 44 in order to maintain a concentration in the gas phase, i.e. in the gas stream to the reactor, of from 0.5% to 5% carbon dioxide by volume. This carbon dioxide range has been found to maintain an adequate dissolved carbon dioxide concentration in the slurry, a factor which is important in achieving effective leaching.

The addition of oxygen to the sulphide mineral slurry 20 is controlled in order to maintain the minimum dissolved oxygen concentration in solution at a value of from 0.2×10^{-3} kg/m^3 to 4.0×10^{-3} kg/m^3. The upper threshold value depends on the genus and strain of microorganism used in the bioleaching process and typically is in the range of from 4×10^{-3} kg/m^3 to 10×10^{-3} kg/m^3.

Figure 1 illustrates the addition of oxygen from a source 32 of pure oxygen. The pure oxygen can be mixed with air from the source 36. Any other suitable gas can be used in place of the air. The addition of oxygen to air results to what is referred to in this specification as oxygen enriched gas ie. a gas with an oxygen content in excess of 21% by volume. It is possible though to add oxygen substantially in pure form directly to the slurry. As used herein pure oxygen is intended to mean a gas stream which contains more than 85% oxygen by volume.

The temperature in the bioleach reactor or vessel may be controlled in any appropriate way using techniques which are known in the art. In one example the tank 18 is insulated and heating takes place by means of energy which is released by the oxidation of sulphides. The temperature of the slurry 20 is regulated using an internal cooling system 70 which includes a plurality of heat exchanger cooling coils 72 connected to an external heat exchanger 74.

The vessel 18 may be substantially sealed by means of a lid 80. Small vents 82 are provided to allow for the escape of off-gas. The off-gas may, if required, be captured or treated in any appropriate way before being released to atmoshpere. Alternatively, according to requirement, the tank 18 may be open to atmoshpere.

The microorganisms chosen for the leaching process will determine the leaching temperature, and vice versa. The applicant has found that a preferred operating temperature is above 60°C, for example in the range of 60°C to 85°C. In this range thermophilic microorganisms, in any appropriate combination, are employed. In the range of from 45°C to 60°C, on the other hand, moderate thermophiles are employed while at temperatures below 45°C mesophiles are used. These microorganisms may, for example, be chosen from those referred to hereinbefore.

Although the benefit of adding oxygen to the slurry which is to be leached, by making use of oxygen enriched air or, more preferably, by making use of substantially pure oxygen ie. with an oxygen content in excess of 85%, is most pronounced at high temperatures at which greater leaching rates are possible, a benefit is nonetheless to be seen when oxygen enriched air or substantially pure oxygen is added to the slurry at lower temperatures, of the order of 40°C or even lower. At these temperatures the leaching rates are slower than

at elevated temperatures and although an improvement results from using oxygen enriched air the cost thereof is generally not warranted by the relatively small increase in leaching rate.

Test Results

The importance of maintaining an adequate supply of oxygen and hence a sufficiently high dissolved oxygen concentration to sustain microorganism growth and mineral oxidation is shown in the results presented in Figure 2. If the dissolved oxygen is allowed to drop below 1.5 ppm, and particularly below 1.0 ppm, biooxidation becomes unstable, which is indicated by higher iron(II) concentrations in solution, of greater than 2 g/l. At consistent levels of biooxidation, achieved by maintaining a dissolved oxygen concentration above 1.5 ppm, in this experiment, iron(II) is rapidly oxidised to iron(III), and iron(II) concentrations remain generally below 1.0 g/l.

The results presented in Figure 2 were obtained from operation of a first or primary reactor of a continuous pilot plant treating a chalcopyrite concentrate at a feed solids concentration of 10% by mass and a temperature of 77°C, with *Sulfolobus*-like archaea.

The effect of increasing the oxygen content of the feed gas to a bioreactor and controlling the dissolved oxygen concentration, in accordance with the principles of the invention, was tested in an experiment using a $5m^3$ bioreactor which was operated with a continuous pyrite or blended pyrrhotite and pyrite flotation concentrate feed, at a temperature of about 77°C, using a mixed culture of *Sulfolobus*-like archaea and a solids density of 10% by mass. The carbon dioxide content in the bioleach inlet gas was controlled at a level of between 1 and 1.5 % by volume. The dissolved oxygen concentration was generally within the range 0.4×10^{-3} kg/m^3 to 3.0×10^{-3} kg/m^3. The results of the experiment are presented in Figure 3.

From the graphs presented in Figure 3 it is clear that, when sparging with air (enriched with carbon dioxide: 20.7% oxygen and 1.0% carbon dioxide), the maximum oxygen demand (directly proportional to the sulphide oxidation duty) was limited to 11.3 kg/m^3/day, since the dissolved oxygen concentration which was achievable using air only (i.e. not enriched with oxygen) was just sufficient to maintain microorganism growth.

By controlling the oxygen content of the inlet gas, the oxygen addition rate, and the dissolved oxygen concentration in the slurry in the range of 0.4×10^{-3} kg/m³ to 3.0×10^{-3} kg/m³, the oxygen demand, i.e. the sulphide mineral oxidation rate, was increased dramatically. The dissolved oxygen concentration was controlled to a low value, but above the minimum limit for successful microorganism growth, so that the utilisation of oxygen was maximised. The results show the oxygen demand, or sulphide oxidation duty, was increased by over threefold. Thus by increasing the oxygen content in the inlet gas from 20.7% to a maximum of 90.8% the specific oxygen demand was increased from 11.3 kg/m³/day to 33.7 kg/m³/day. In addition, by controlling the dissolved oxygen concentration to a low value, but above the minimum value for sustained microorganism growth, the oxygen utilisation was maximised. The oxygen utilisation showed a general increase with an increase in the oxygen content of the inlet gas from 29% (for an inlet gas oxygen content of 20.7%) to 91% (for inlet gas containing 85.5% oxygen).

The high oxygen utilisations achieved of well over 60% are much better than expected. Analysis of the results indicates that the oxygen mass transfer coefficient (M), as defined by equation (1), is significantly and unexpectedly enhanced for operation of the bioreactor at a high temperature (77°C) and with a high oxygen content in the inlet gas (from 29% to 91% in the experiment). In fact, the oxygen mass transfer coefficient (M) is increased by a factor of 2.69, on average, compared to the applicant's design value. This enhancement is after considering the improvement in the mass transfer coefficient due to temperature, which would be expected to increase the value of M by a factor of 1.59 for a temperature increase from 42°C to 77°C, according to the temperature correction factor proposed by Smith et al [5]. This correction factor has been demonstrated experimentally to be valid for a temperature in the range of from 15°C to 70°C [6].

The determination of the enhanced oxygen mass transfer coefficient is shown from the results presented in Figure 4, where the oxygen demand divided by the design oxygen mass transfer coefficient (M_{design}) is plotted against the oxygen driving force, as defined in equation (1). The slope of the regression line plotted through the data indicates the enhancement in the oxygen mass transfer coefficient by a factor of 2.69.

Process Examples

The inventive principles in the preceding section have been described in the context of sulphide minerals in general and, as will be appreciated by those skilled in the art, can be applied to copper bearing sulphide minerals in particular.

Figure 5 of the accompanying drawings is a process flow chart illustrating one form of the method of the invention for recovering copper.

In Figure 5 the plant 10 which is shown in Figure 1 and which is described hereinbefore bears the same reference numeral. The oxygen and carbon dioxide sources respectively bear the reference numerals 32 and 34. The copper bearing sulphide slurry is labelled with the numeral 50.

The flow sheet in Figure 5 is an example of the invention applied to copper bearing sulphide minerals, arsenical sulphides such as enargite, as well as copper bearing sulphide minerals which are refractory to mesophile leaching, such as chalcopyrite.

Copper bearing sulphide concentrate slurry 50 is leached in the plant 10 which contains one or more bioleach reactors, using oxygen enriched gas or substantially pure oxygen 32 as the oxidant. The oxygen concentration in the reactor is controlled in a manner which has been described hereinbefore depending on the type of microorganism used. The plant 10 produces a bioleach residue slurry 100 which contains solubilised copper, and iron predominantly in the ferric state.

If the copper bearing sulphide concentrate 50 contains arsenical copper sulphide minerals such as enargite then the bioleach residue 100 will contain solubilised arsenic. In this instance the residue 100 is subjected to a liquid-solid separation step 102 producing solids 104 for disposal and solution 106, which is fed to a pH adjustment step 108 in which the pH of the solution is adjusted by the addition of limestone 110, resulting in partial iron removal by precipitation. Arsenic which is present in the slurry is also precipitated.

Carbon dioxide 114 produced in the step 108 may be fed to the slurry in the plant 10 by being blended with oxygen from the source 32 or with carbon dioxide from the source 34, or by being injected directly into the slurry in the plant.

Slurry 116 produced by the step 108 is returned to the main flow line.

If the ferric iron-to-copper ratio in solution is unfavourable for solvent extraction it may also be desirable to carry out the step 108 directly on the bioleach residue 100.

The residue 100, or the slurry 116, as the case may be, is then subjected to a liquid/solid separation step 118 producing solids 120 for disposal, and a solution 122. The solution in turn is fed to a solvent extraction step 124. Strip liquor 126 from the solvent extraction step is obtained by stripping the loaded solvent with spent electrolyte 128 from a copper electrowinning step 130 which produces copper metal cathodes 132. Oxygen gas 134 generated at the anode in the electrowinning process is fed to the source 32 to supplement the supply of oxygen to the plant.

Raffinate 136 from the solvent extraction step 124 is neutralised (138) by the addition of limestone 140 and the resulting slurry 142 is disposed of. A portion of the raffinate may optionally be recycled to the bioleach step 10 or, if appropriate, to an external heap leach 144, to satisfy acid requirements of these process.

Optionally, if there is insufficient carbonate in the slurry 50, carbon dioxide 146 which is generated in the neutralisation step 138 may be fed to the slurry in the plant 10 eg. by being blended with the gas stream from the source 32 or by being added to the carbon dioxide source 34.

Figure 6 illustrates another example of the invention wherein a pre-leaching step is applied to a copper bearing sulphide concentrate. Arsenical copper sulphide such as enargite can also be handled in the manner shown in Figure 6.

The bioleaching plant 10 of Figure 1 again bears the reference numeral 10 in Figure 6 and the oxygen and carbon dioxide sources respectively bear the reference numerals 32 and 34.

Copper bearing sulphide concentrate 150 may be pre-leached in one or more pre-leach reactors 152 using a stream 154 of an acidic solution of copper and ferric sulphate which is produced in a manner described hereinafter.

A slurry 156 produced by the pre-leaching stage 152 is then subjected to a liquid/solid separation step 158 producing residue solids 160 which are fed to the bioleaching plant 10, and a solution 162.

If the concentrate 150 contains arsenical copper sulphide minerals such as enargite then the solution 162 and stream 154 each contain solubilised arsenic. In this instance the solution 162 is fed to a pH adjustment step 164 in which the pH of the solution is raised by the addition of limestone 165 and results in iron removal by precipitation. Arsenic which is present in the slurry is also precipitated.

A slurry 166 emerging from the step 164 is then subjected to a liquid/solid separation step 168 producing solids 170 for disposal and a solution 172 which is returned to the main flow line.

If the solution 162 and stream 154 do not contain arsenic then the solution 162 is fed to a solvent extraction step 173, which is described later.

The residue slurry 160, which contains non-reacted copper and other sulphide minerals, is leached in the plant 10 which contains one or more bioleach reactors using oxygen enriched gas or substantially pure oxygen 32, as the oxidant, in the manner which has been described hereinbefore. The oxygen concentration in the reactor is controlled to a suitable value, depending on the type of mircroorganism used.

The bioleaching process produces a bioleach residue slurry 174 which contains solubilised copper and iron predominantly in the ferric state.

The bioleach residue 174 is subjected to a liquid/solid separation step 176 producing solids 178 for disposal and the solution 154 of solubilised copper and iron which is used in the pre-leaching stage 152.

The solution 162 is fed to the solvent extraction step 173. Strip liquor 190 from the solvent extraction step is obtained by stripping the loaded solvent with spent electrolyte 192 from a copper electrowinning step 194 which produces copper metal cathodes 196. Oxygen gas 198 generated at the anode during the electrowinning process is directed to the slurry in the plant 10, for example by being added to the gas stream from the oxygen source 32.

Raffinate 200 produced during the stage 173 is neutralised (202) with limestone 204 and the resulting slurry 206 is disposed of. A portion of the raffinate may optionally be recycled to the bioleach plant 10 or, if available, to an external heap leach 208 to satisfy acid requirements of those processes. Carbon dioxide 210 produced in the neutralisation step 202 may be directed to the slurry in the plant 10, for example by being added to the gas stream from the oxygen source 32 or by being added to the carbon dioxide from the source 34. Carbon dioxide 212 produced in the step 164 may be similarly handled.

Particular Example

Bioleach pilot plant test work was completed, using a chalcopyrite concentrate assaying 32% copper (75% chalcopyrite), on a ~1.1 m^3 pilot plant consisting of 6 reactors configured as 2 primary reactors in parallel followed by 4 secondary reactors in series. The total primary volume was 470l and the total secondary volume was 630l. All test work was carried out at a temperature of from 77°C to 80°C using a feed slurry containing 10% solids. The microorganisms used were a mixed *Sulfolobus*-like archaea. The oxygen utilisation results obtained in the primary stage during the test work, using analysis of inlet and outlet gas mixtures, are shown in Table 3.

Table 3 : Primary Reactor Copper Dissolution and Oxygen Uptake Results for Thermophile Pilot Test Work

Retention Days	Cu Dissolution %	Specific Cu Dissolution Rate $kg/m^3/h$	Oxygen Uptake (calculated) $kg/m^3/h$	Oxygen Uptake (measured) $kg/m^3/h$
2.8	60.5	0.312	0.668	0.638
2.4	55.9	0.336	0.718	0.704

Minimal chalcopyrite leaching, possibly reaching 35% copper dissolution, is found to occur at 40°C using mesophiles.

CLAIMS

1. A method of recovering copper from a copper bearing sulphide mineral slurry which includes the steps of:

(a) subjecting the slurry to a bioleaching process.

(b) supplying a feed gas which contains in excess of 21% oxygen by volume. to the slurry. and

(c) recovering copper from a bioleach residue of the bioleaching process.

2. A method according to claim 1 which includes the step of pre-leaching the slurry prior to the bioleaching process of step (a).

3. A method according to claim 2 wherein the pre-leaching is effected using an acidic solution of copper and ferric sulphate.

4. A method according to any one of claims 1 to 3 which includes the step of removing ferric arsenate from the bioleach residue before step (c).

5. A method according to claim 4 wherein the ferric arsenate is removed by precipitation.

6. A method according to any one of claims 1 to 5 wherein the bioleach residue is subjected to a neutralisation step which produces carbon dioxide which is fed to the feed gas of step (b) or directly to the slurry.

7. A method according to any one of claims 1 to 6 wherein in step (c) copper is recovered using a solvent extraction and electrowinning process.

8. A method according to claim 7 wherein oxygen generated during the copper electrowinning is fed to the feed gas of step (b). or directly to the slurry.

9. A method according to claim 7 or 8 wherein raffinate, produced during the solvent extraction, is supplied to at least one of the following: the bioleaching process of step (a), and an external heap leach process.

10. A method according to any one of claims 7 to 9 wherein oxygen generated during the electrowinning process is fed to the feed gas of step (b) or directly to the slurry.

11. A method according to any one of claims 1 to 10 wherein the said slurry contains at least one of the following: arsenical copper sulphides, and copper bearing sulphide minerals refractory to mesophile leaching.

12. A method according to claim 11 wherein the said slurry contain chalcopyrite concentrates.

13. A method according to any one of claims 1 to 12 wherein the feed gas contains in excess of 85% oxygen by volume.

14. A method according to any one of claims 1 to 13 which includes the step of maintaining the dissolved oxygen concentration in the slurry within a desired range.

15. A method according to claim 14 wherein the said dissolved oxygen concentration is maintained in the range of from 0.2×10^{-3} kg/m^3 to 10×10^{-3} kg/m^3.

16. A method according to any one of claims 1 to 15 which includes the step of controlling the carbon content of the slurry.

17. A method according to any one of claims 1 to 16 which includes the step of controlling the carbon dioxide content of the feed gas in the range of from 0.5% to 5.0% by volume.

18. A method according to any one of claims 1 to 17 which includes the step of bioleaching the slurry at a temperature in excess of 40 °C.

19. A method according to claim 18 wherein the said temperature is in the range of from 40°C to 100°C.

20. A method according to claim 19 wherein the said temperature is in the range of from 60°C to 85°C.

21. A method according to any one of claims 1 to 17 which includes the step of bioleaching the slurry at a temperature of up to 45°C using mesophile microorganisms.

22. A method according to claim 21 wherein the microorganisms are selected from the following genus groups: *Acidithiobacillus: Thiobacillus; Leptosprillum; Ferromicrobium;* and *Acidiphilium.*

23. A method according to claim 21 or 22 wherein the said microorganisms are selected from the following species: *Acidithiobacillus caldus (Thiobacillus caldus); Acidithiobacillus thiooxidans (Thiobacillus thiooxidans); Acidithiobacillus ferrooxidans (Thiobacillus ferrooxidans); Acidithiobacillus acidophilus (Thiobacillus acidophilus); Thiobacillus prosperus; Leptospirillum ferrooxidans; Ferromicrobium acidophilus;* and *Acidiphilium cryptum.*

24. A method according to any one of claims 1 to 17 which includes the step of bioleaching the slurry at a temperature of from 45°C to 60°C using moderate thermophile microorganisms.

25. A method according to claim 24 wherein the microorganisms are selected from the following genus groups: *Acidithiobacillus* (formerly *Thiobacillus); Acidimicrobium; Sulfobacillus; Ferroplasma (Ferriplasma);* and *Alicyclobacillus.*

26. A method according to claim 24 or 25 wherein the said microorganisms are selected from the following species: *Acidithiobacillus caldus* (formerly *Thiobacillus caldus); Acidimicrobium ferrooxidans; Sulfobacillus acidophilus; Sulfobacillus disulfidooxidans; Sulfobacillus*

thermosulfidooxidans: *Ferroplasma acidarmanus*; *Thermoplasma acidophilum*; and *Alicyclobacillus acidocaldrius*.

27. A method according to any one of claims 1 to 17 which includes the step of bioleaching the slurry at a temperature of from 60°C to 85°C using thermophilic microorganisms.

28. A method according to claim 27 wherein the microorganisms are selected from the following genus groups: *Acidothermus*; *Sulfolobus*; *Metallosphaera*; *Acidianus*; *Ferroplasma (Ferriplasma)*; *Thermoplasma*; and *Picrophilus*.

29. A method according to claim 27 or 28 wherein the said microorganisms are selected from the following species: *Sulfolobus metallicus*; *Sulfolobus acidocaldarius*; *Sulfolobus thermosulfidooxidans*; *Acidianus infernus*; *Metallosphaera sedula*; *Ferroplasma acidarmanus*; *Thermoplasma acidophilum*; *Thermoplasma volcanium*; and *Picrophilus oshimae*.

30. A method of bioleaching a slurry containing copper bearing sulphide minerals which includes the steps of:

(a) bioleaching the slurry using suitable microorganisms at a temperature in excess of 40°C, and

(b) controlling the dissolved oxygen concentration in the slurry within a predetermined range.

31. A method according to claim 30 wherein the said dissolved oxygen concentration is controlled by controlling the supply of oxygen to the slurry.

32. A method according to claim 31 wherein the oxygen is supplied to the slurry in the form of oxygen enriched gas or substantially pure oxygen.

33. A method according to any one of claims 30 to 32 wherein the said temperature is in the range of from 60°C to 85°C.

34. A method of enhancing the oxygen mass transfer coefficient from a gas phase to a liquid phase in a copper bearing sulphide mineral slurry which includes the step of supplying a feed gas containing in excess of 21% oxygen by volume, to the slurry.

35. A method according to claim 34 wherein the feed gas contains in excess of 85% oxygen by volume.

36. A method according to claim 34 or 35 which includes the step of raising the temperature of the slurry.

37. A method of bioleaching an aqueous slurry containing copper bearing sulphide minerals which includes the steps of:

(a) bioleaching the slurry at a temperature above 40°C, and

(b) maintaining the dissolved oxygen concentration in the slurry in the range of from 0.2×10^{-3} kg/m^3 to 10×10^{-3} kg/m^3.

38. A method according to claim 37 wherein the dissolved oxygen concentration in the slurry is maintained by supplying gas containing in excess of 21% oxygen by volume to the slurry.

39. A method according to claim 37 or 38 wherein the temperature is in the range of from 60°C to 85°C.

40. A method of recovering copper from a slurry containing copper bearing sulphide minerals which includes the steps of bioleaching the slurry using suitable microorganisms at a temperature in excess of 40°C, controlling the dissolved oxygen concentration in the slurry within a predetermined range, and recovering copper from a bioleach residue.

41. A method according to claim 40 wherein the bioleaching is carried out at a temperature in excess of 60°C.

42. A method according to claim 40 or 41 wherein the dissolved oxygen concentration in the slurry is maintained by supplying gas containing in excess of 21% oxygen by volume to the slurry.

43. A method according to claim 42 wherein the gas contains in excess of 85% oxygen by volume.

44. A plant for recovering copper from a copper bearing sulphide mineral slurry which includes a reactor vessel. a source which feeds a copper bearing sulphide mineral slurry to the vessel, an oxygen source. a device which measures the dissolved oxygen concentration in the slurry in the vessel, a control mechanism whereby, in response to the said measured dissolved oxygen concentration, the supply of oxygen from the oxygen source to the slurry is controlled to achieve a dissolved oxygen concentration in the slurry within a predetermined range, and a recovery system which recovers copper from a bioleach residue from the reactor vessel.

45. A plant according to claim 44 wherein the oxygen source supplies oxygen in the form of oxygen enriched gas or substantially pure oxygen to the slurry.

46. A plant according to claim 44 or 45 wherein the reactor vessel is operated at a temperature in excess of 60°C.

47. A plant according to any one of claims 44 to 46 which includes a pre-leaching stage for leaching the copper bearing sulphide mineral slurry before the slurry is fed to the reactor vessel.

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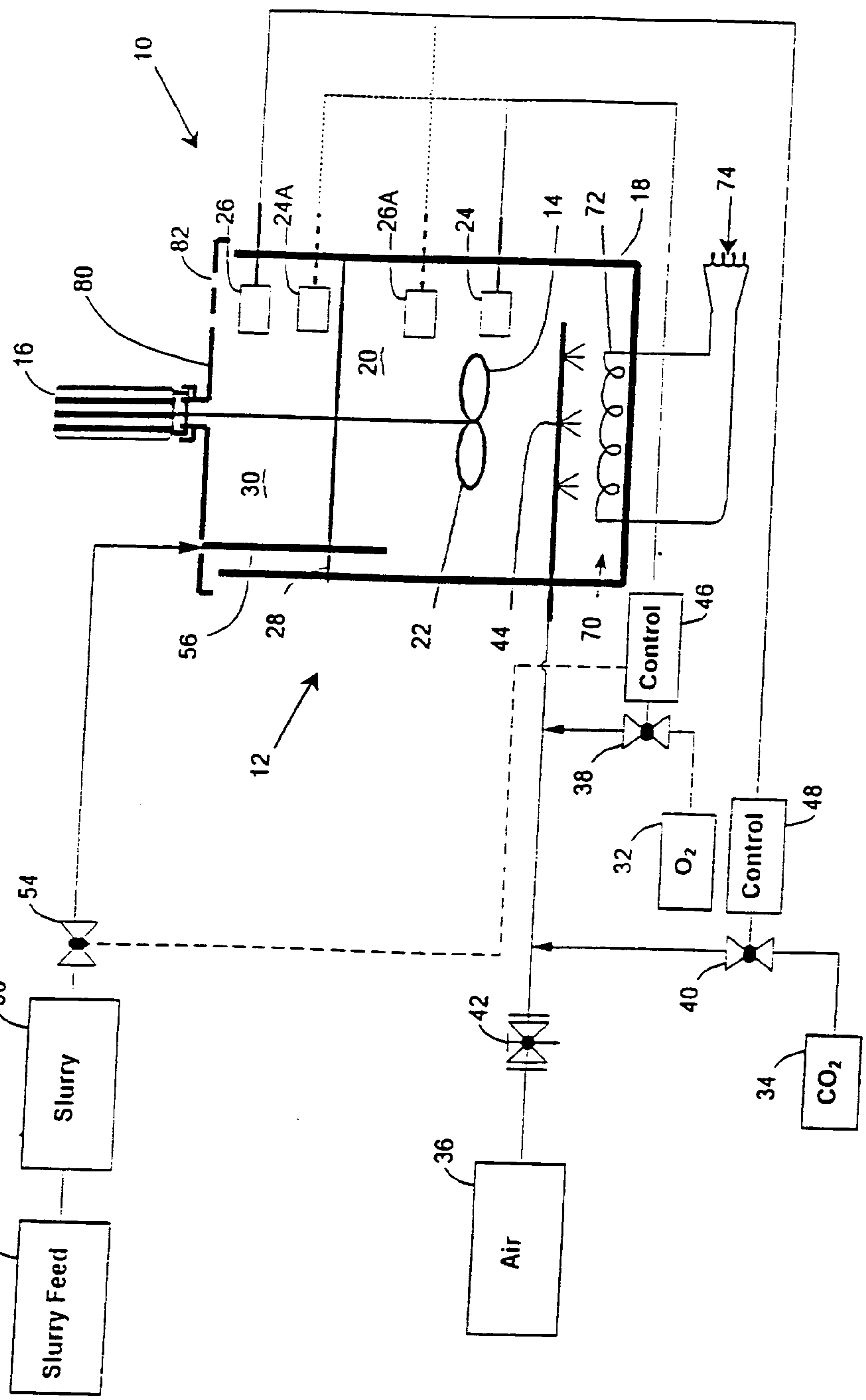
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Control
Control
O_2
CO_2
Air
Slurry
Slurry Feed

Figure 1

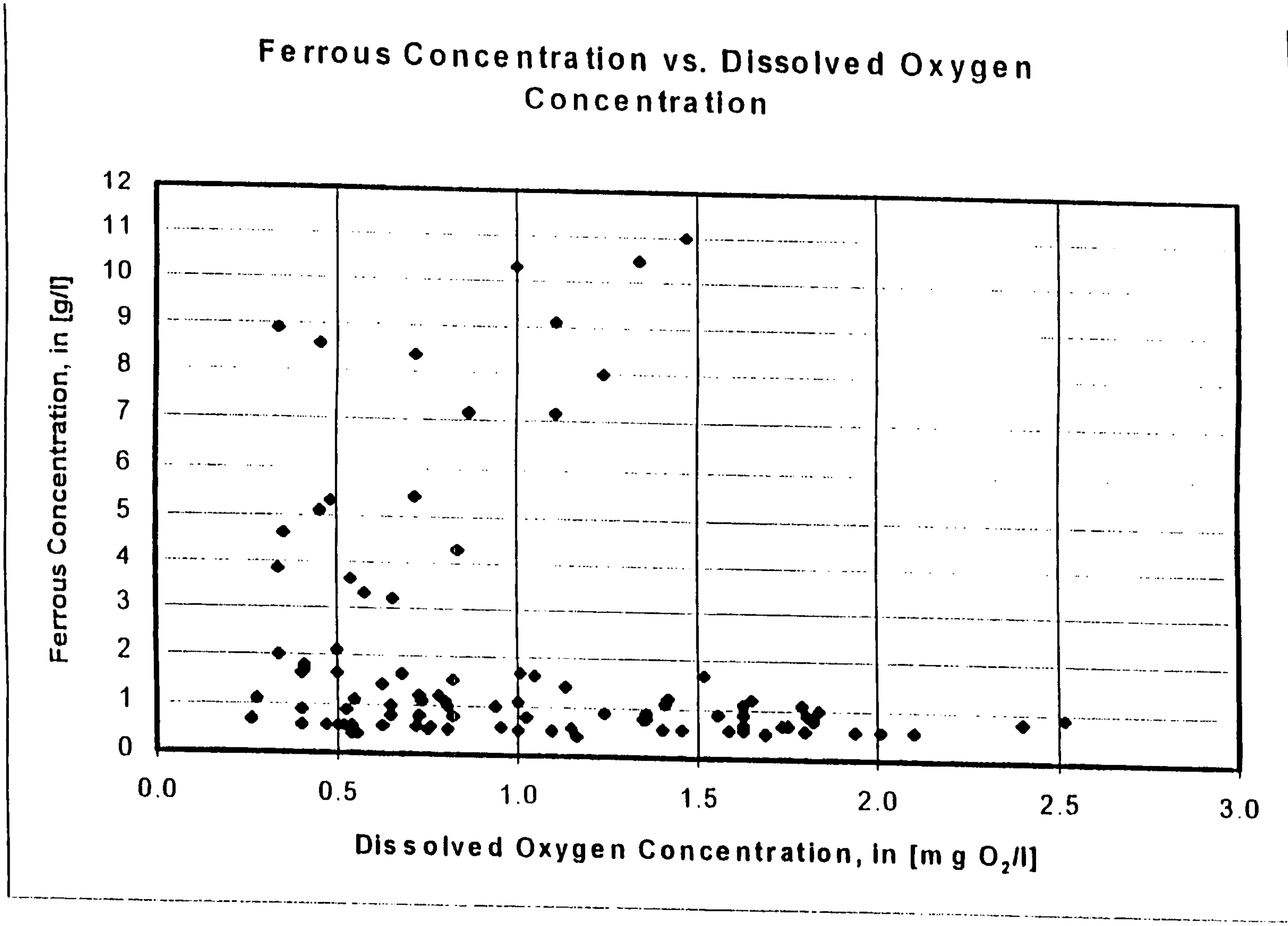

Figure 2 Results Showing The Effect of Reduced Microbial Oxidation On Iron(II) Concentration Levels In Solution As A Result Of Operating At Low Dissolved Oxygen Concentrations

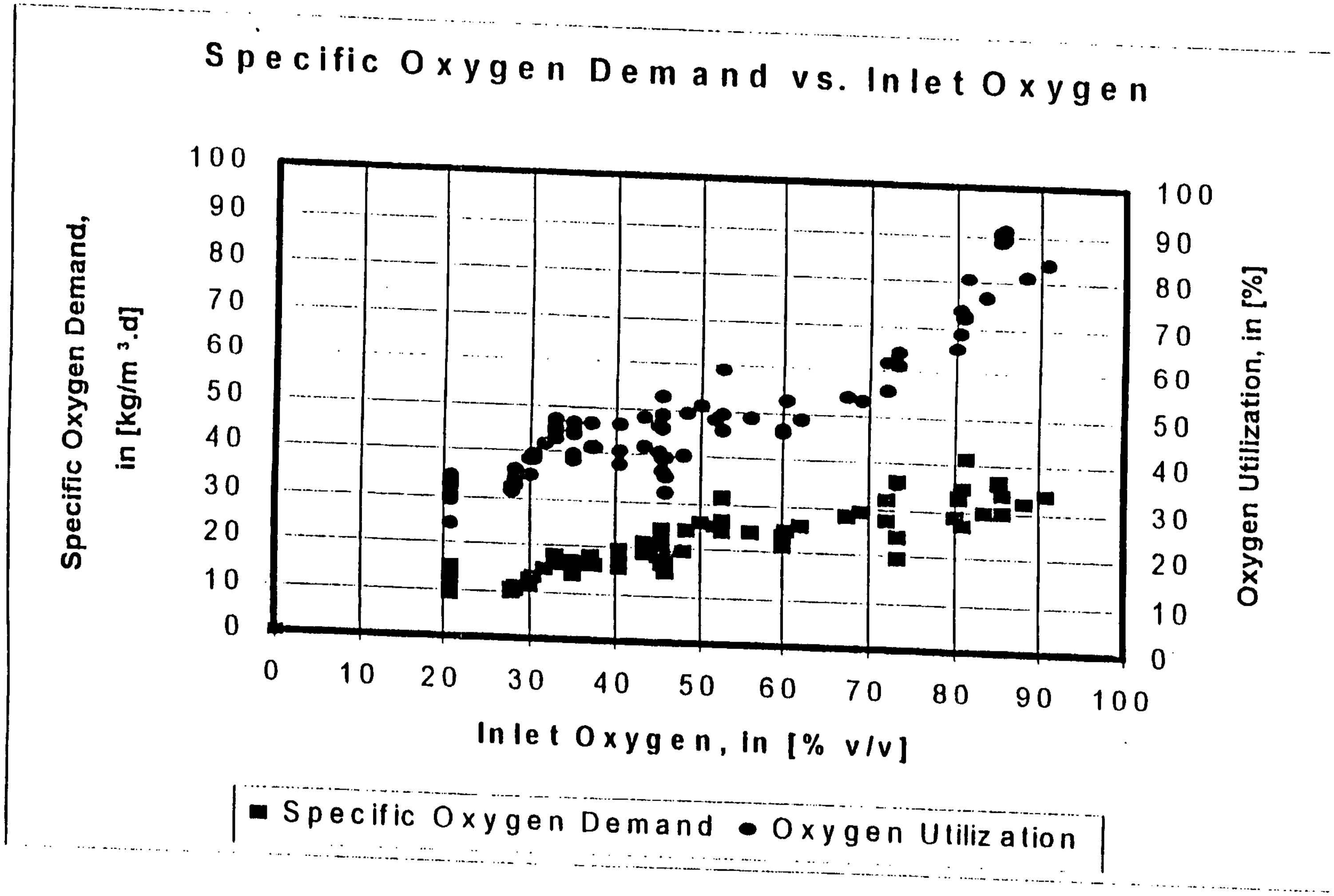

Figure 3 Results Showing The Increase In Specific Oxygen Demand and Oxygen Utilisation Achieved By Increasing The Oxygen Content Of The Inlet Gas Under Controlled Conditions By The Method Of The Invention

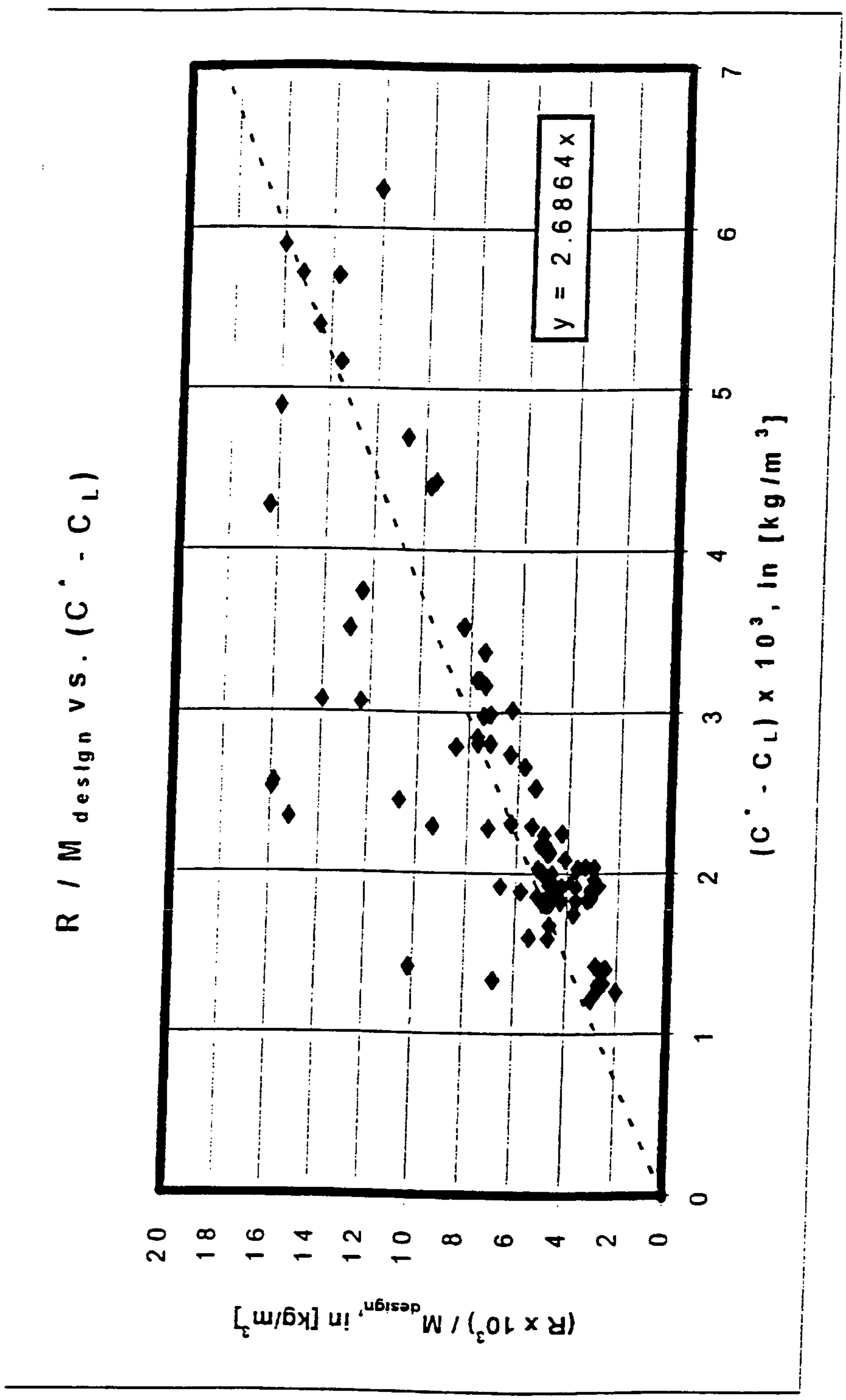

Figure 4 Results Demonstrating The Enhancement Of The Oxygen Mass Transfer Coefficient

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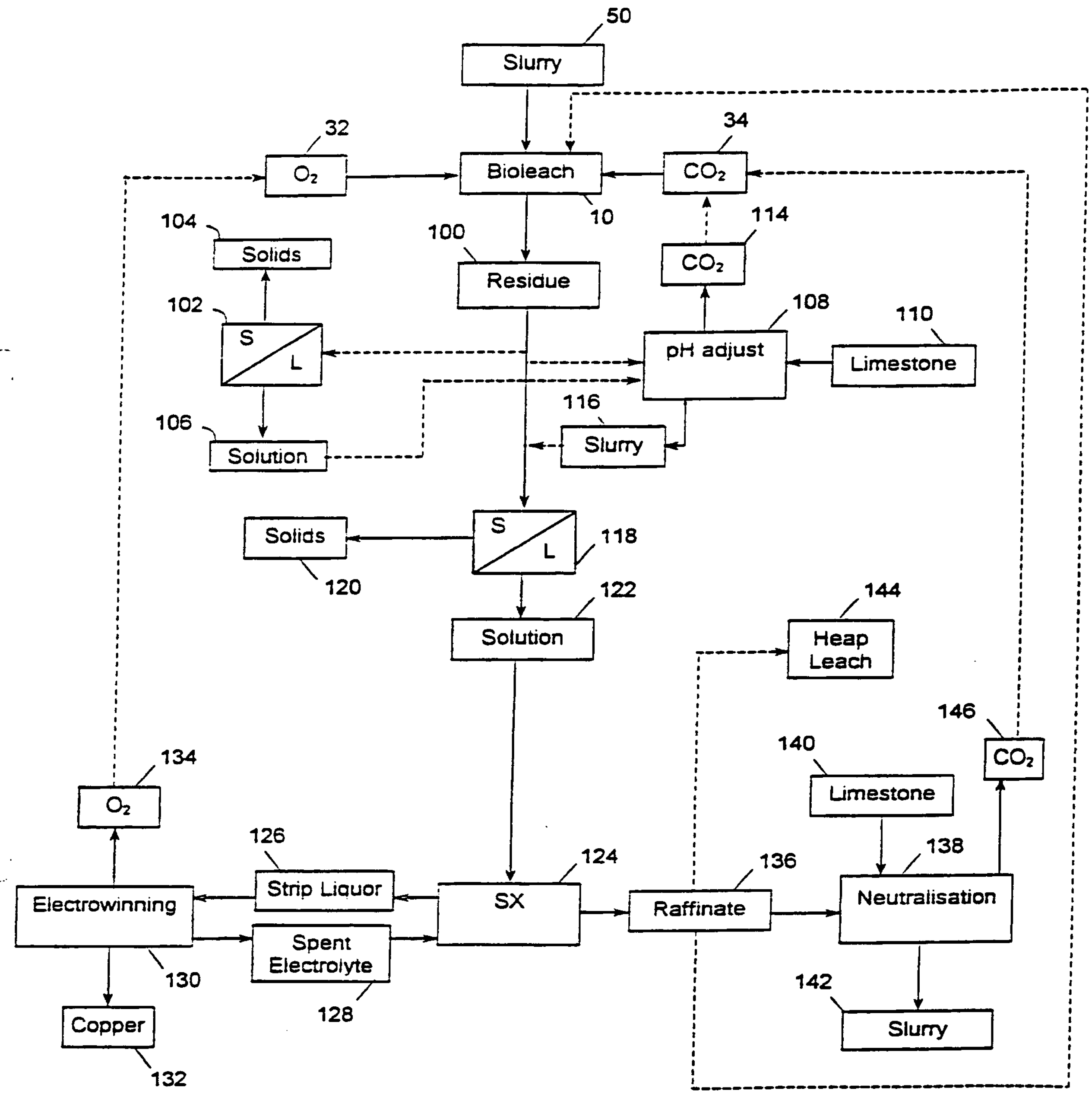

Figure 5

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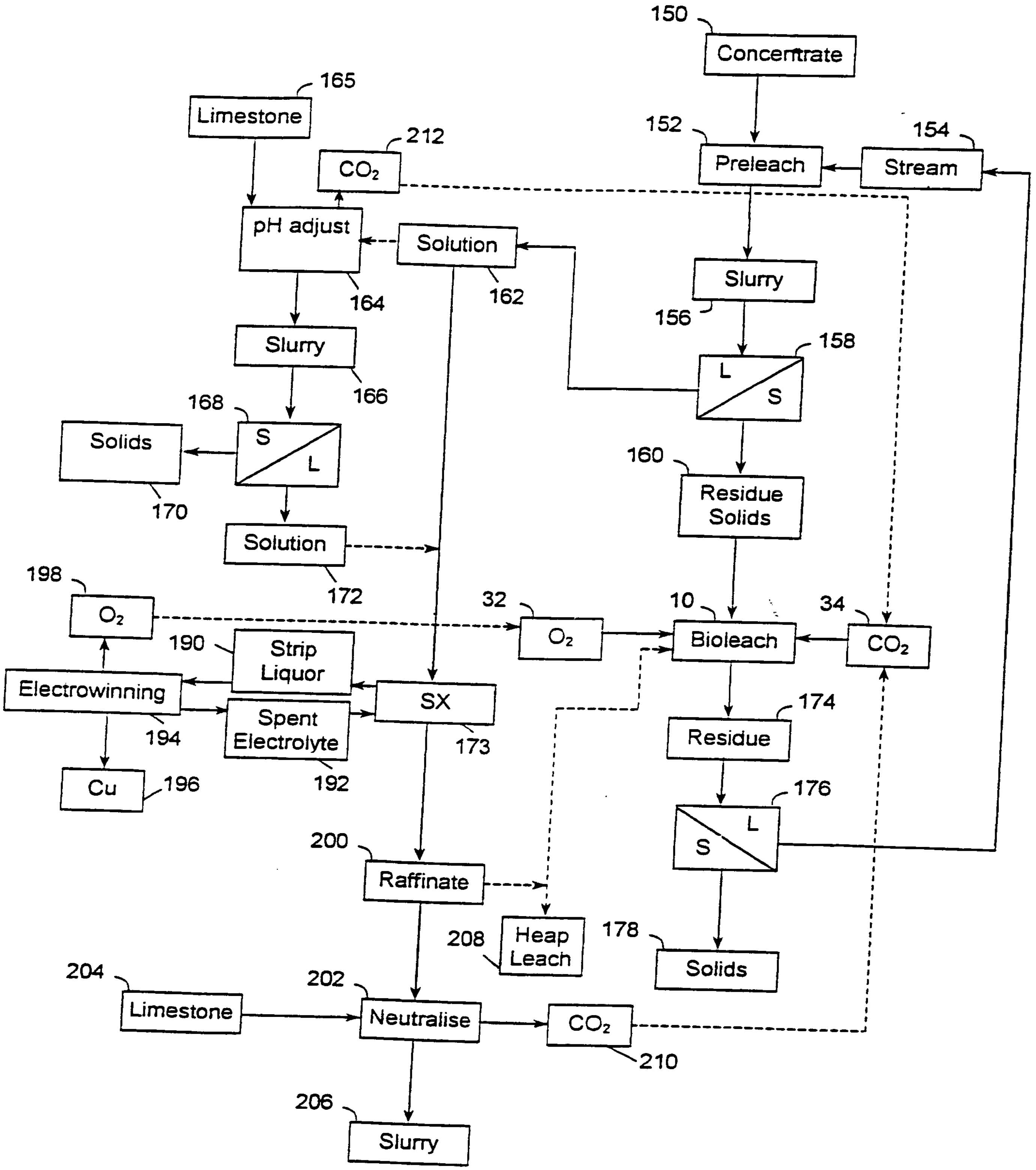

Figure 6